# The Journal

of the

# SOCIETY OF DYERS AND COLOURISTS

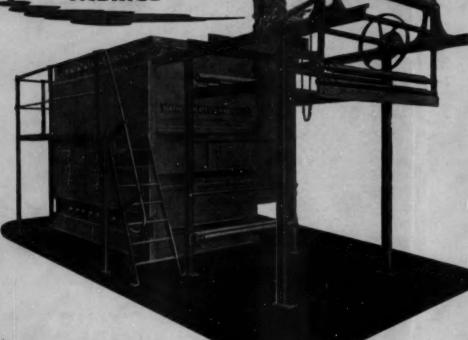
Volume 70 Number 8

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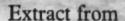
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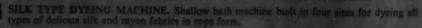
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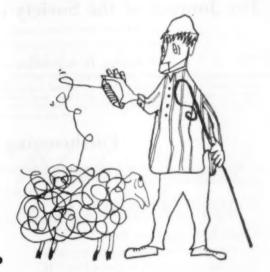


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(Abstracts section only printed on one side of paper—£1 10s 0d per annum)

## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1954 and pages 262–266 of the July 1954 issues of the January 1954 and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138–9). Editorial Communications should be addressed to The Editor, at the same address.

# Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the Journal —

The Application of Resins to Textiles

LECTURES
A. R. Smith

## COMMUNICATIONS

A Study of Certain Natural Dyes - I and II

The Polarography of Azo Dyes

Researches upon the Light Fading of Dyes - II

Dyeing of Celloluse Acetate with Non-ionic Dyes - I and II

The Combination of Keratin with Acids

Some Factors in the Measurement of Affinities of Vat Dyes for Cellulose

F. M. Arshid, C. H. Giles, et al. J. de O. Cabral and H. A. Turner

H. R. Chipalkatti, C. H. Giles, et al.

T. G. Majury

L. Peters

R. H. Peters and J. Simons

### F.T.C.C. PUBLICATION

The Use of Fading Lamps for Determining Light Fastness

K. McLaren





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## FORTHCOMING MEETINGS OF THE SOCIETY

Wednesday, 15th September 1954

MIDLANDS SECTION. Knitwear Miscellany. A. W. Carpenter, Esq., P. Gildesgame, Esq., R. M. Gimson, Esq., J. A. Ridge, Esq., with H. W. Hitchman, Esq., (Chairman). (Joint meeting with Hinckley Textile Society) Masonic Hall, Hinckley. 7.15 p.m.

Tuesday, 28th September 1954
Scottish Section. Is Dyeing Theory of any use to the Practical Dyer? Dr. L. Peters (Lecturer in Textile Industry Leeds University). St. Enoch Hotel, Glasgow.

Friday, 1st October 1954

LONDON SECTION. The Wear Properties of Resin Treated Fabrics. Dr. T. H. Morton (Courtaulds Ltd.). Royal Society Rooms, Burlington House, London, W.1. 6 p.m.

Wednesday, 6th October 1954
MIDLANDS SECTION. The Dyeing of Wool at High Temperatures. D. R. Lemin, Esq., B.Sc., H. Sagar, Esq., A.T.I., F.I.C., and G. A. Coutie, Esq., B.A., (Mr. Lemin lecturing). College of Technology, Leicester. 7 p.m.

Wednesday, 13th October 1954

NORTHERN IRELAND SECTION. Economic, Technical and Other Difficulties in Meeting Fastness Requirements. S. Burgess, Esq. (Courtaulds Ltd.). Thompson's S. Burgess, Esq. (Courtaulds Ltd.). Th Restaurant, Donegall Place, Belfast. 7.30 p.m.

Tuesday, 19th October 1954

Scottish Section. Some Notes from a Rayon Piece Dyer's Note-book. C. C. Wilcock, Esq., A.R.T.C.S., F.T.I., F.S.D.C. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow. 7 p.m.

Thursday, 28th October 1954

MIDLANDS SECTION. Discussion on Dyeing Machine
Design. Opening Speaker — F. H. Marsh, Esq., B.Sc.,
F.R.I.C. Gas Theatre, Nottingham. 7 p.m.

Friday, 5th November 1954

LONDON SECTION. Unserviceable Fabrics from the Dry Cleaner's Viewpoint. Exact title later. Mr. Moulder (Dyers and Cleaners Research Organisation) (Joint Meeting with London Centre of the Guild of Dyers and Cleaners). Royal Society Rooms, Burlington House, London, W.1. 6 p.m.

Wednesday, 10th November 1954

NORTHERN IRELAND SECTION. Some Observations on American Bleaching and Dyeing Processes. J. K. Skelly, Esq. (Belfast College of Technology). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Tuesday, 16th November 1954
SCOTTISH SECTION. Silicones in the Taxtile Industry. T. W. Watson, Esq., B.Sc. (Midland Silicones Ltd.). St. Enoch Hotel, Glasgow. 7 p.m.

Wednesday, 17th November 1954

MIDLANDS SECTION. Dyeing of Terylene above 100°C.

A. S. Fern, Esq., B.Sc., F.S.D.C. Kings Head Hotel, Loughborough. 7 p.m.

Friday, 19th November 1954

MIDLANDS SECTION. Colour fastness from the point of view of the Manufacturer, Dyer and Consumer (Forum) (Joint meeting with the Leicester Textile Society and Textile Institute). College of Technology, Leicester. 7 p.m.

MIDLANDS SECTION. LADIES EVENING—DINNER DANCE. Black Boy Hotel, Nottingham. 7 p.m.

Wednesday, 1st December 1954

MIDLANDS SECTION. The Evaluation of Optical Bleaching Agents. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint meeting with the British Association of Chemists). College of Art, Derby. 7 p.m.

Friday, 3rd December 1954
LONDON SECTION. The Dyeing of Textile Fibres at Temperatures above 100°C. Mr. J. A. Fowler (I.C.I. Ltd.).
Royal Society Rooms, Burlington House, London, W.I. 6 p.m.

Tuesday, 7th December 1954

Scottish Section. Electrical Aids to Production in the Textile Industry. L. Landon Goodman, Esq., B.Sc. (British Electrical Development Association). St. Enoch Hotel, Glasgow. 7 p.m.

Wednesday, 8th December 1954

NORTHERN IRELAND SECTION. Some aspects of Design in Bleaching and Finishing Plant. W. W. Spooner, Esq. (Spooner Dryer and Engineering Co. Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 6th January 1955

NORTHERN IRELAND SECTION. The Modern Approach to Linen Bleaching and Finishing I. M. Hill, Esq. (Milltown Bleaching Co. Ltd.) (Joint lecture with Textile Institute). Thompson's Restaurant, Donegall Place, Institute). Thomp Belfast. 7.30 p.m.

Friday, 7th January 1955

LONDON SECTION. Details later.

Wednesday, 12th January 1955

NORTHERN IRELAND SECTION. Application and Evaluation of Optical Bleaching Agents. Messrs. G. Taylor and A. V. Cole (Clayton Dyestuffs Co. Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Wednesday, 26th January 1955
MIDLANDS SECTION. 1 do it this way! Demonstrations of novel testing techniques by local members. College of Technology, Leicester. 7 p.m.

Friday, 4th February 1955
LONDON SECTION. Evaluation of Textiles. Exact title later.
J. S. Ingham Esq. (Marks & Spencer Ltd.). Royal Society Rooms, Burlington House, London, W.1. 6 p.m.

Wednesday, 9th February 1955
NORTHERN IRELAND SECTION. Fastnesses of Coloured Textiles as they Affect the General Public. J. S. Ingham Esq. (Marks & Spencer Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Wednesday, 23rd February 1955

MIDLANDS SECTION. The Classification of Dyestuffs by their
Dyeing Characteristics. W. Beal, Esq., B.Sc. Kings Head
Hotel, Loughborough. 7 p.m.

Friday, 4th March 1955
MIDLANDS SECTION DINNER.
Kings Head Hotel, Loughborough. 7 p.m.

Wednesday, 9th March 1955

NORTHERN IRELAND SECTION. Film Evening. (Joint Meeting with Textile Institute). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 10th March 1955

MIDLANDS SECTION. The Diffusion of Vat Dyes into Cellulose.
R. H. Peters, Esq., Ph.D., F.S.D.C., and T. Watson,
Esq., Ph.D. (Dr. Peters lecturing). Gas Theatre, Nottingham. 7 p.m.

Wednesday, 6th April 1955
MIDLANDS SECTION. Paper by G. H. Lister, Esq., B.Sc.,
Ph.D. (Subject later) (ANNUAL GENERAL MEET-ING at 6.30 p.m.). Kings Head Hotel, Loughborough.

Thursday, 21st April 1955

MIDLANDS SECTION. Some Experiences of a knitted-goods dyer. A. W. Carpenter. (Joint Meeting with the Textile Institute Kidderminster Section). Carpet Trades Canteen, Mill Street, Kidderminster. 7 p.m.



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## THE JOURNAL

OF THE

# Society of Dyers and Colourists

Volume 70 Number 8

AUGUST 1954

Issued Monthly

## Proceedings of the Society

THE ELEVENTH JOHN MERCER LECTURE

## Physics in the Dyeing, Printing, and Finishing Industries

N. W. YELLAND

Meeting held at the Midland Hotel, Manchester 2, on 8th April 1954, Mr. F. L. Goodall (President of the Society) in the chair

The applications of physics in textile processing are reviewed, with particular reference to calico printing. Among topics discussed are colour, lustre, drape and handle, thermal and electrical properties, measurement of moisture content, repeating patterns, optical measurements, engraving, and microscopy.

John Mercer was born in 1791, two years after Antoine Lavoisier had published his renowned treatise on chemistry 1; Mercer's work on the application of chemistry to calico printing was begun in 1812 2, four years after John Dalton had announced his atomic theory 3; thus Mercer lived and worked throughout an era of rapid expansion of the science of chemistry, namely the first half of the nineteenth century.

The occasion of the present lecture suggests that it would be interesting to trace the parallels between Mercer's achievements and the application of physical research to the same industries which has been made in the first half of the twentieth century, at a time when the science of physics, in its turn, has been in course of an intense development.

But if the lecture is to be kept to a reasonable length, the suggested theme can be treated only by means of illustration, and the general character of the impact of the science on the industries can perhaps be displayed in an interesting way by describing a few typical instances in which physical research, method, or inventions have found useful applica-An objection concerns the limitations of individuals: it is unlikely that any one person could give a complete and balanced account of the subject. In this connection, I have to point out that my own experience has been chiefly in the field of the cellulosic fibres; thus in so far as this discourse will be illustrated by reference to firsthand experience with particular fibre substances, there is going to be too much in it about cellulose and its derivatives. The only consolation I can offer is that, if this talk had chanced to be given on the other slope of the Pennines, there would perhaps have been too much in it about the protein fibres.

There are certain broad subdivisions of the field which may first be noted—

- (1) The study of the physical properties of the textile products en masse in their finished or semi-finished state, namely those properties of the material which directly determine their usefulness or their æsthetic appeal. This study in many cases requires and includes the invention of methods, apparatus, and machines for the testing of the physical properties.
- (2) The investigation of the fundamental properties of textile fibres, and of the materials of which they are composed, which determine their behaviour in the various finishing processes which are applied to them, and in their ensuing history of use and wear.
- (3) The study, measurement, and control of the physical factors which govern the operation of finishing processes.
- (4) The invention of new machines and processes, including an activity which consists in keeping under review the current progress of discovery and invention in pure and applied physics with the object of bringing to bear on textile finishing practice those innovations which can profitably be adapted and applied there.

Having drawn up the above list, and having compared it with what I know of the occupations of physicists in the industry over a period of years, I realised that the list is not all-inclusive, and that it is necessary to add a fifth category—

(5) Miscellaneous.

In what follows, an account will be given of some typical applications, arranged more or less in accordance with the five categories which have been listed.

#### COLOUR

The physical properties of a finished textile material which decide its appearance are often those which matter most. Of these factors of appearance, the colour is usually the most significant, and is the one upon which dyers and printers expend the greater part of their effort. It might therefore be expected that the scientific study of colour would be a subject to which they attend with close and continuous interest.

On the scientific side, the principles of colour and of its exact measurement have been studied for many years, a variety of instruments for the latter purpose are available, and international agreement has been reached, between the principal standardising laboratories of the world, concerning the system through which the physical measurements on a sample shall be translated so as to indicate the character of the colour sensation experienced by a normal "standard observer" who sees the sample under a specified illuminant. Thus it might appear that conditions are favourable to the widespread adoption of precise methods of colour measurement in industry generally and especially in the industries that we are considering.

The applications of colour measurement to problems of the dyeing and printing industries have been admirably illustrated in two papers which have been read to this Society 4.5. As suggested in these lectures, and as is well known in the trade, the use of the methods and instruments of exact colour measurement is not at all widespread in the industry.

For their part, physicists are disposed to take for granted the dictum of their exemplar, Lord Kelvin, who said 6—

When you can measure what you are speaking about, and express it in numbers, you know something about it, and when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meagre and unsatisfactory kind. It may be the beginning of knowledge, but you have scarcely in your thought advanced to the stage of a science.

But the prevailing state of affairs, in relation to the measurement of colour in textile dyeing and printing, might lead one at first sight to question whether Lord Kelvin's doctrine applies in that field. Is colour perhaps regarded as an example of a quality, not a quantity? Are the rules which govern its measurement (more strictly its specification) thought to be essentially different from those which apply to the measurement of recognised physical quantities like length, mass, volume, energy, etc., or even temperature? The latter comparisons are in any case inexact, for, since three independent co-ordinates are required for the complete specification of a colour, its measurement is analogous to specifying the position of a point in space by three independent measurements at right-angles, and the colour measurement may be thought of as defining the exact "location"

of the colour, in relation to other colours, in a three-dimensional map (or globe) or "colour-space". The latter can be represented by actual three-dimensional solid models in ordinary space, as shown by Vickerstaff and White 4,5, for example.

Colour measurement would obviously find several applications in these industries: for example, in measuring the colours obtained in routine production, so as to control the effect of fluctuations in production variables, in evaluating with useful precision the results of trial dyeing and printing experiments, in deciding whether colours obtained in production are within tolerance limits of variation, in measuring the durability of colours in fading tests, and generally in keeping under close observation and exact control what is after all the most important feature of the product of the industries.

Nevertheless, the number of dyeworks or printworks which make use of colour measurement is probably small in relation to the total. The reasons are of interest: they have to do partly with the science of colour measurement itself (as expressed by available methods and apparatus), and perhaps partly also with the climate of opinion in the industry.

There is considerable activity in the design of new instruments and methods, and in their application to particular problems, as witness recent reviews of the subject 7.8, but the principal instruments commercially available for general work are still of two types only, namely spectrophotometers, for the spectrum analysis of fundamental colour properties, and colorimeters, for the specification of colour appearances. While both types of instrument are necessary in a central or standardising laboratory in which colour is studied, both types have their disadvantages, and no available instrument of either type is ideally suited for industrial work of the sort which I have been considering. It may be of interest to state what seem to be the requirements of a colour-measuring instrument for the latter work-

- It should be of the colorimeter type, for speed and directness.
- (2) The sensitive detector of the instrument should be a photocell instead of the eye, though on this point reference may be made to a fairly recent note by W. D. Wright \*9.
- (3) The precision of measurement should be of the order of 0.1% rather than the 1% which represents the best that can be consistently obtained if the eye is used as a balance detector for brightness or chromaticity.
- (4) The instrument should indicate the colour properties of the sample as they would appear to the Standard Observer of the international system (C.I.E.).
- (5) The readings of the instrument should be convertible quickly and easily into terms of scales of visually uniform steps (of hue, saturation, and lightness) like the scales of the Munsell system for example, or an agreed "uniform chromaticity scale".

(6) The instrument should be of reasonable simplicity and cheapness, and should be rapid and straightforward in operation.

(7) As a relatively minor practical point, which is nevertheless important and sometimes over-looked by instrument designers, the instrument should be easily applicable to the types of sample that are of interest, namely dyed or printed textile materials, in large or small areas, either in the piece or as small samples cut from the piece.

It is not known that any colorimeter answering fully to such requirements is yet available. A note on this point is included in Wright's paper <sup>9</sup>. A good account of types and makes of instrument available up to 1945 is supplied by a report of the Committee on Colorimetry of the Optical Society of America <sup>10</sup>.

Thus it appears that one of the reasons why colour measurement is not applied more extensively is that no instruments for the purpose are yet available which are completely satisfactory in all respects: there is room for development and improvement of instruments and methods. It is of interest to note, however, that in the past, improvements in this field have hardly ever originated with the instrument manufacturers, but with academic or industrial scientists, in short not with the instrument-makers but with the instrument-users. At present, intensive work is still going on, at every level of fundamental study, of human colour vision and of practical application to industrial colour problems. But already so much is known, concerning the main facts and concerning the requirements and the possibilities of the situation, that something might be gained if the instrument-makers were to take a broader view of their function, and to consider what contribution they could usefully make.

The deficiencies of available instruments may not be the only factor. Is it also possible, for example, that the principles of colour physics are not widely or deeply understood within the colour industries, or by those who would have most occasion to apply this science? Is it the case that the methods of colorimetry do not form any important part of the courses of technical or academic instruction designed for those who are to become colour technologists? It seems to me that these are questions that might profitably be considered by this Society in the context of its plan to establish academic training and recognised qualification in colour technology.

I suggest that the science of colour measurement could be (but is not) to the colourist what the science of navigation is to the mariner: in both arts the desired objective can be represented by a point or small region in a three-dimensional space. Is it as important for a colourist to arrive at the correct colour of a dyeing as it is for the mariner to make a good landfall?

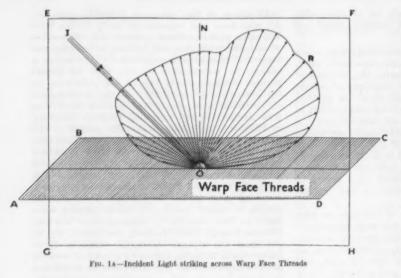
White and Vickerstaff have noted, in their Mercer lecture for 1945 5, that the intensity of interest in problems of colour science is much lower in this country than it is in the United States, as witness, for example, the output of

publications in the principal scientific journals. All who have had occasion to study in this field will agree with the above authors, and will wonder what is the price that will have to be paid for our neglect of this avenue of advance. Are we, as a nation, to recapitulate in the field of colour physics the melancholy tale of our abdication in the field of colour chemistry! In the development of synthetic dyes, important pioneer work was done in this country, following Perkin's discovery in 1856, but leadership eventually passed to the German chemists and industrialists, who were able to create, and to maintain until 1914, a virtual monopoly in this field and in related fields of synthetic chemistry. All that is well known, but is it equally well known that a parallel development is taking place in our own time? Fundamental studies in colour vision and colour physics were made in this country by pioneers like Newton, Thomas Young, Clerk Maxwell, and Abney, but in our time the ripened technical value of their discoveries is being harvested by a band of workers in the United States. The full effect no doubt lies in the future, but the consequences are already being felt in some measure, for example in the fields of colour cinema and colour television. The most advanced systems of colour television are of American origin; it appears likely that if we in this country should eventually enjoy (if that is the right word) the benefits of colour television (whatever these may be), we shall pay tribute in dollars for the privilege.

#### LUSTRE

Another set of optical properties of a finished textile which are of interest are those which govern its lustre. In a mercerised finish on cotton, for example, it is requisite that the lustre be as high as possible and silk-like in character. In a schreinered finish the lustre should also be high, and should have a particular sort of silk-like quality different from glaze or polish. But again, in certain finishes, a high glaze or polish is actually required, while in the finishing of certain continuous-filament rayon fabrics, which normally may have a high inherent lustre, it is in some instances desired to reduce the lustre by means of a delustring treatment.

The measurement of lustre is of value in studying and controlling the operation of the various processes which are employed. It is known that lustre-appearance is determined chiefly by the manner in which light is reflected from the cloth, particularly upon the extent to which light-rays are reflected specularly, as they would be from a mirror, or, on the other hand, are scattered in all directions, as they would be in reflection from a matt surface like blotting paper, for example. In reflection from a smooth structureless glossy surface, the degree of specular concentration is high for all directions in the surface, but in the case of lustrous woven textiles, the degree of specular concentration is, typically, high when the light-rays fall along the predominant surface threads, but much lower for light-rays which strike across those threads (Fig. 1).



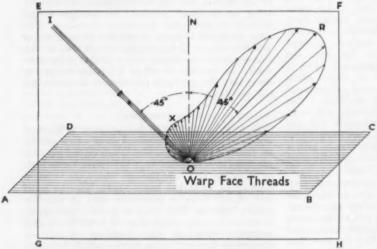


Fig. 18-Incident Light striking along Warp Face Threads

AB ... DC Warp Face Threads EFHG Plane of incidence

Fig. 1—Lustre Characteristic Curves. Angular Distribution (Polar Diagrams) of Reflected Light for a Light Ray incident at 45° on a Lustrous Satin Cloth. (In the polar diagrams the length of each reflected ray is drawn proportional to the intensity of light reflected in that direction)

Methods of lustre measurement depend in some way upon measuring the degree of specular concentration for rays which fall across the cloth surface in various directions in relation to the surface threads. A typical instrument for the purpose comprises an arrangement rather like a spectrometer or crystal goniometer, in which a flat specimen of the sample can be rotated on every possible axis through measured angles and can be illuminated at various angles by a parallel beam of light, and the intensity of reflected light can be measured on a suitable photometer or reflectometer, which may be either visual or photoelectric. Such an instrument is illustrated in Fig. 2.

The results obtained on these instruments are usually expressed in the form of "characteristic curves", which are found by making measurements of reflection intensity as the angular relationships of the goniometer head are varied point after point in some prescribed way. The shape and the height of such curves give a good deal of information about the character and the intensity of the lustre. For example, a highly lustrous fabric in one method of testing gives a sharply peaked curve. If the cloth is smooth, like a satin, the peak is single; if the cloth is ribbed, like a poplin, the peak is duplex (Fig. 3-5).

Instead of plotting lustre curves by the rather laborious method of point-by-point measurement, it has also been found possible to record them directly by using a photoelectric cell to pick up the reflected light, and displaying its amplified output directly upon the screen of a cathode-ray oscillograph, as shown in Fig. 6–8.

John Mercer's name is commemorated in the title of a famous process for improving the lustre of cotton, and it is apropos to mention that the methods of lustre measurement, which I have been describing, have been much used in the study and control of the mercerising process.

THE "DRAPE" AND THE "HANDLE" OF TEXTILES

The physical properties of a finished textile which determine its draping

quality, and the feel of it in the hand, are in many cases next in importance to those which govern its appearance. Of these physical properties, the most important in this connection are a group of mechanical factors, viz. the stiffness, the roughness, and the softness or compressibility. Appropriate methods for the evaluation of these properties have been developed.

A valuable account of this subject was given in the well known paper by F. T. Peirce, on "The Handle of Cloth as a Measurable Quantity" <sup>11</sup>. The apparatus and the methods which Peirce recommended for this study are of remarkable simplicity and economy, and the required measurements are such as anyone can make for himself;

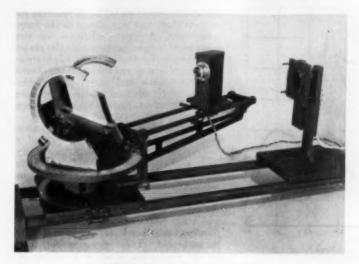


Fig. 2— Goniophotometer for Lustre Measurement

yet, by his mathematical analysis of the governing factors, Peirce showed how it is possible to calculate, from these very easy measurements, constants which will accurately indicate the relative values of all the factors which govern the relative "handle" and "draping" qualities of any series of

This work has withstood the test of time. In a recent paper 12, the conclusion is stated that the Peirce method is as reliable as certain alternative methods, and six times as fast. Although much other work has been reported on this subject, the work of Peirce stands as a good and typical example of the application of the methods of physics to the study of the mechanical properties of textiles. In a similar way, methods have been established for the measurement of most of the mechanical mass properties of textiles which have a recognised commercial or industrial significance.

#### THERMAL PROPERTIES

Among the thermal properties of finished textile piece goods, those which determine thermal insulating value are of most interest, for example in relation to protection against heat loss in the case of wool blankets or garment materials, or in protection against solar radiation in the case of certain cotton cloths. Measurement of heat insulation value was obviously useful, and became possible as soon as thermometry had become sufficiently accurate to permit reasonably good measurements of thermal conductivity and surface emissivity. We find accordingly that quantitative studies of thermal insulation began in about 1880. They are still continuing, using the improved methods of measurement that are available today, and are in some

instances being applied to newly devised materials. Thermal insulation is perhaps of more interest to the wool manufacturing industry than to those which work with other fibres; nevertheless this problem has been studied by physicists working in relation to nearly all the fibres which are of commercial importance 13,14,

## ELECTRICAL PROPERTIES

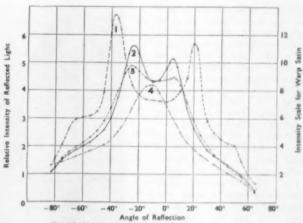
Since textile materials, for example cotton cloth, tapes, and threads, are used for electrical insulation, their electrical resistance is an important feature, and in that application it is desired to make the resistance as high as possible. On the other hand, since the resistance can under some conditions be very high, difficulties are

sometimes caused, in finishing processes, by static electrification of the material, which cannot be discharged because of the high resistance. These problems present an obvious challenge to the physicist; studies are continuing, and a measure of understanding and practical success has already been achieved.

It is interesting to note that the problem of static electrification of textile materials is being successfully attacked by the use of ionising radiations from artificial radioactive substances manufactured by neutron irradiation in the atomic piles of the nuclear energy research stations.

#### THE PROPERTIES OF TEXTILE FIBRES

There are many fundamental physical properites of textile fibres which are of the greatest importance in textile manufacture and finishing-



Highly lustrous artificial silk poplin

Highly lustrous cotton poplin Cotton poplin, same cloth as (2) but less lustrous finish Mercerised warp satin

Fig. 3 — Typical Characteristic Curves showing Difference between Satin Lustre and Poplin Lustre

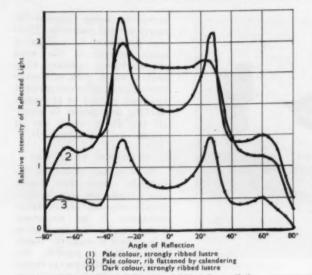


Fig. 4 - Lustre Differences of Ribbed Rayon Cloths

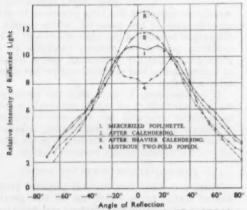


Fig. 5 — Set of Characteristic Curves showing the Effect of Calendering on the Lustre of Mercerised Poplinette Cloth

e.g. the length and the fineness of the fibres, their shape of cross-section, their stiffness, their tensile strength and hardness, their surface smoothness, polish, and frictional properties, their interior structure, their surface character, their degree of crystallinity and molecular orientation, their absorbency for water and dyes. All these have been studied; these studies are all of interest and have led to conclusions which have practical significance. There is, however, space only to mention a single fibre property, and I have chosen one that has not yet been mentioned, namely plasticity.

Plasticity is the opposite of elasticity. A perfectly elastic substance may be deformed by

the application of force, but it recovers its original shape and size as soon as the force is removed. A plastic substance, on the other hand, retains permanently the shape into which it is deformed by the application of force. The material of most textile fibres is partly elastic and partly plastic; the balance of plastic and elastic properties profoundly affects their behaviour in every stage of manufacture and use.

A discussion of the technical significance of the plasticity of textile fibres and a description of a method for its exact study were given by Peirce in 1923 15. Peirce showed how the plasticity could be measured by the rate of decay of shear stress produced in the material, and he described a magnetic torsiometer with which the plasticity of single fibres, fibre bundles, and other forms of material could be measured. These measurements lead to the evaluation of a few constants by which the plastic behaviour of the material can be characterised. Since 1923 much work has been done on rheology in general, and on the plastic flow properties of textile materials, but I am not aware that any of this has affected the lasting value of Peirce's work.

While most of the desired qualities of finished textiles can be studied by the measurement of the appropriate physical properties of the material, some of the processes employed in finishing involve chemical reactions, in which physical factors like temperature, pressure, velocity, etc. may play an important part. But there are many instances in which the process itself is also wholly physical in character, e.g. mangling, calendering, schreinering, and drying. The last is,

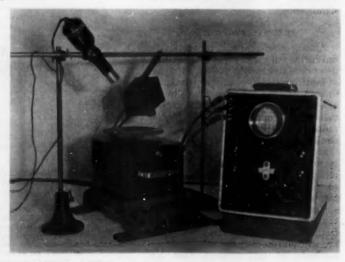


Fig. 6 - Apparatus for Cathode-ray Oscillograph Display of Lustre Characteristic Curves

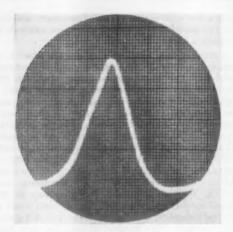


Fig. 7 — Cathode-ray Oscillograph Lustre Curve for Satin Weave

perhaps, a good example to consider, since most textile piece goods, of whatever fibre, are saturated with water at some stage of finishing, and have to be dried again.

Studies of drying are naturally directed towards obtaining the maximum efficiency, speed, and convenience in the operation of drying machinery. We notice immediately that the idea and the definition of efficiency prove elusive in this connection. Many drying machines are operated by condensing steam in them, and using the latent heat of the steam to evaporate water from the cloth. This is true, for example, of both candrying ranges and hot-air drying stenters. It is an obvious suggestion to measure the efficiency of a drying range by the weight of water evaporated from the cloth when unit weight of steam is condensed in the machine or when the equivalent amount of heat is supplied in some other way—

# Machine efficiency = $\frac{\text{Weight of water evaporated}}{\text{Weight of steam condensed}}$

If one pound of water is evaporated when one pound of steam is condensed, the efficiency would be in the region of 100%. Now, while it is true that, other things being equal, a machine with a high efficiency may be better than one with a low efficiency, yet it is also true that efficiency as so defined has not the same significance as the familiar idea of the efficiency of a heat-engine. The latter cannot be more than 100% efficient, but drying efficiency can be much more than 100%, depending on how the denominator of the above fraction is measured. Consider the domestic washing-day. Millions of pounds of water are evaporated from textiles every Monday, and if you have to enter in the above fraction any measure whatever of the heat expended, you enter in the denominator a zero: the efficiency is infinite. The heat of evaporation comes from the atmosphere, but nobody has to pay for it. Drying by that means is, however, slow; in industrial drying we supply energy in some form and in some way, in order that the cloth may be dried quickly, and in a machine that is reasonably compact. Thus the performance of a

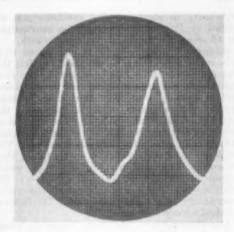


Fig. 8—Cathode-ray Oscillograph Lustre Curve for Poplin Weave

drying machine is not usually to be judged by the efficiency figure alone, but the capital cost of the machine, and its installation and factory space costs, must also be taken into account.

Can-drying ranges are very widely used, and have much in their favour, including a useful capacity for self-regulation. The appetite of the machine for steam depends on how much work it is doing. The temperature of the hot surface cannot rise above the boiling point of water at the supply pressure; thus overheating of the cloth is automatically prevented, though overdrying may occur if the cloth remains stationary on the cans. There appears to be little scope for the improvement of can-drying machines, except by the use of efficient cylinder-buckets to minimise the film of condensed water on the inside wall of the can, and by maintaining a reasonably low humidity in the air-space outside the cans.

In stenter drying machines, the possibility of controlling the speed and the efficiency of drying appears to be somewhat greater, since the speed, the temperature, and the humidity of the air circulated for drying can all be regulated independently, and a definite problem can be formulated, viz. for any given task of drying, what is the most economical combination of temperature, speed, and humidity? What are the best proportions of power consumption between heaters and fans? What is the best proportion of recirculated air? Would it be economic to pass the moisture-laden air, which has passed over or through the cloth, through a heat-exchanger in which it gives up its heat to incoming dry air, and keeps the evaporated water out of circulation? In the latter connection, what is the value of water vapour itself as a medium for conveying heat to the cloth? What, if any, are the advantages of using superheated steam (i.e. water vapour at a pressure lower than its saturation pressure) for drying? All these problems are in a field which is thoroughly familiar to the physicist, and in which the methods of physics can and have been applied with success. For example, the distribution of air-velocity in stenter drying machines can be studied by Pitot-tube surveys, using instruments like those employed in aerodynamic research.

Machines for drying cloth at high speed tend to be rather long; this is because heat to effect the evaporation can as a rule be supplied only at a limited rate per unit area, so that an extended time of passage is needed. If the rate of heat transfer per unit area could be increased, the machines could be made shorter. The increase in rate of energy transfer can be obtained by using radiant heat, as in infrared drying machines. The temperature of the radiator is so regulated that the energy is radiated most strongly at the wavelengths which are strongly absorbed by the cellulose—water complex; if this is done, the rate of transfer of energy can be very high. The basis in physical science is obvious.

Experiments have also been made in which energy is transferred by the power loss in the material from a high-frequency electrical current. This method has the advantage that the energy transfer continues at a much reduced rate after the water has been evaporated, so that overheating is minimised. The disadvantage is the high cost of the large thermionic valves which at present have to be employed.

In the case of infrared driers, there is no such automatic safeguard against overheating, and safety devices must be installed to cut off the heat in the case of a halt of the cloth in the drier.

# MEASUREMENT OF THE MOISTURE CONTENT OF RUNNING CLOTH

The moisture content of cloth as it passes through the machines has to be controlled. This is the case with some of the drying machines which have been mentioned, in order to prevent damage by overdrying. But for other reasons, also, it is necessary to control the moisture content with some accuracy. For example, in processes such as calendering, schreinering, and embossing, which depend on the plasticity of the material, the moisture content must be closely controlled. Also moisture control was found to be of crucial importance in certain finishing processes which require the fixation of synthetic resins in the fibre. Methods of obtaining an indication of the moisture content of textile webs moving through the machines have been studied, for example, at the British Cotton Industry Research Association. It was known that the electrical conductivity of cellulosic fibres is extremely sensitive to moisture content, and a range of appliances were developed for using this fact to measure moisture content accurately.

These appliances were entirely suitable for use in the processes of many of the firms within the research association, and were widely adopted. It is, however, not to be expected that such apparatus would meet the needs of every possible problem of that kind; for example, dyers, printers, and finishers often process cloth which contains a proportion of electrolytes sufficient to lower the electrical resistance by a large and fluctuating amount. The electrical resistance is

then a less convenient index of moisture content, and the question of possible alternative methods of indication arises.

A number of alternative methods are possible in principle. Instead of a measurement of electrical resistance, the cloth can be passed between the plates of an electrical condenser through which a high-frequency alternating current is passed, and the indication of moisture content is then based upon the power loss of the current in the material. In another possible method, the cloth can be passed through a small chamber in which air at a controlled humidity is circulated, and two sets of electrodes are arranged differentially to measure the electrical resistance of the cloth entering, and leaving, the test chamber. A signal may be obtained according as the cloth gains or loses water in the test chamber: if it loses, it is too wet; if it gains, it is too dry. This method utilises the very high sensitivity of the resistance method, eliminates the disturbing effect of contained electrolytes, and refers directly to a humidity standard which can be verified continuously or at any moment.

Other types of method can be based upon the moisture condition of air in equilibrium with the cloth. A sheet of moving cloth draws a layer of air along with it, and in the absence of disturbing factors, the air close to the cloth may be in moisture equilibrium with the cloth. Thus the air humidity will be high if the cloth is wet, and low if the cloth is dry. Within certain limits the moisture content of the cloth-can be deduced from the air humidity; on the other hand, in certain cases the equilibrium humidity may be of more direct significance than the actual moisture content. The air humidity can be indicated in various ways: for example, the air-stream may be made to flow over the surface of a dew-point hygrometer consisting of a polished metal plate, the full width of the cloth, in which a temperature gradient is established in a direction perpendicular to the width. The moist air will condense on the plate at a height limit dependent on the humidity; the height of the edge of the mist-patch indicates the dampness of the cloth at any point across its width. In connection with such projects, there is obviously wide scope for the employment of the methods of physics in the study of the velocity, temperature, and humidity of the entrained air-stream, and in the design of the apparatus in detail.

Fig. 9 shows a primitive form of apparatus that was used in early work on this subject. The air scoop is a sheet-metal triangle in plan. The air enters by a narrow slit which may be the full width of the cloth, and the scoop tapers to a few inches in plan width at the vertical pipe, in which the air flows past a hair-hygrometer and then past a wet-and-dry bulb hygrometer, and to an exhaust fan with a speed regulator. In another form of the apparatus, the vertical pipe and the fan were not used: the entrance scoop was balanced by an exactly similar suction scoop joined on to it, and the hygrometer element was placed at their junction. Even with such crude apparatus as this, it is possible to establish that a useful correlation

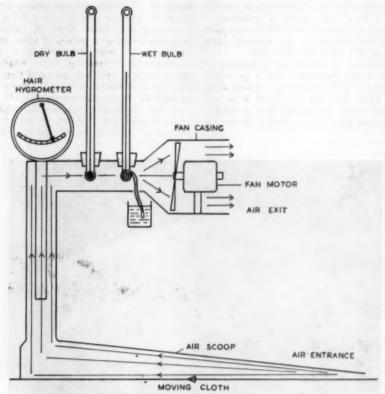


Fig. 9 - Air-sampling Scoop for Cloth Dryness Testing

exists, under the appropriate conditions, between the moisture condition of the cloth and the hygrometer readings. In subsequent developments, the whole apparatus is based on steady-flow principles, an electrical hygrometer is used to give a direct indication of moisture content, and by means of a system of electronic valve amplifiers and relays, the operation of the drying machines is controlled automatically so as to hold the moisture content of the material close to any desired value.

#### DIMENSIONS OF RUNNING CLOTH

It is usually desirable that the finished cloth be dimensionally stable, and not subject to shrinkage or stretch during use and laundering. Since cloth has to be put in tension to draw it through the machines, it generally tends to stretch somewhat at that stage, and is left with some tendency to shrink lengthwise when subsequently released. Various finishing processes exist for the purpose of counteracting this effect, and ensuring that the cloth remains stable at the dimensions at which the customer receives it. In the operation of any of these processes it is necessary to know the conditions of lengthwise stretch at which the running cloth exists in the machines; this information is most conveniently expressed by the number of transverse (weft) threads per unit length of the cloth. Since the

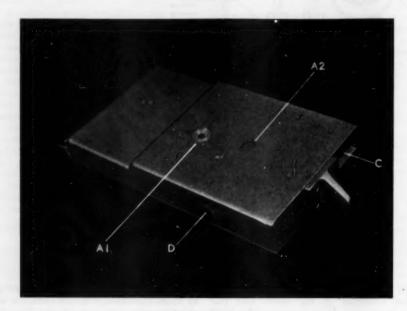
cloth is moving continuously, it is not practicable to count the threads in the ordinary way with a counting glass, or taper grid. This problem is soluble in a variety of ways. If stroboscopic illumination of a correct frequency is used, the taper grid pattern becomes visible again, and can be used in cases where the required precision is not very high. If the cloth is brightly illuminated by transmitted light, and an image of the cloth is formed on a slit parallel to the weft, each weft thread as it passes produces a signal in the form of a pulse of light. These pulses can be counted by means of a photocell, amplifier, and electronic counter of the type which has been developed primarily for particle counting in nuclear physics. Suitable discriminators from the same field are also available for converting the rather irregular pulses from the threads into pulses that are sufficiently regular for counting. At ordinary speeds of running, the number of pulses per second is of the order of a thousand, which is easily within the counting rate of modern counters. The counting is made to yield the required information by arranging that it is automatically confined to a specified length of material, e.g. 10 in. or 100 in. of cloth, the cycle of counting and reading being continuously repeated. Other methods, based on the use of light pulses and photocells, have also been employed; there seems to be no doubt that most counting or cloth-length problems of this

kind could be solved by the application of similar methods; e.g. a very high degree of precision is possible by using a photocell detector to compare the spacing of the threads in the moving cloth with the spacing of a fixed standard grid.

The above example illustrates an application of electronics; the latter technique has a powerful fascination because of its versatility. Cases arise from time to time, however, in which it is not particularly convenient to employ any available form of apparatus which incorporates electronic valves.

has some disadvantages and some sources of error. There is consequently the need for a method of measuring the total dose of light-action administered during a period of time, regardless of variations of intensity. What is required is evidently an integrating meter which will total the product of light-intensity and time in the same way as a domestic electricity meter totals the product of electric current and time <sup>17</sup>.

Several integrating meters of this type are known commercially, but they are all rather bulky and expensive. It is desirable that the light-sensitive



- A I Light-aperture for photocell
- A 2 Light-aperture for neon tube
- Counter window
- D Desiccator container

Fig. 10-Compact Light Integrator for Fading Lamps

#### PHOTOELECTRIC LIGHT INTEGRATOR

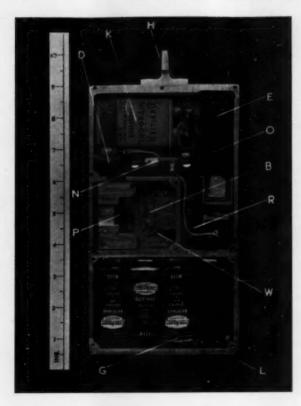
The Subcommittee of your Society which is considering the methods that are employed for testing the fastness of dyeings to light has cooperated with the British Standards Institution in issuing a specification which recognises daylight as the only standard of illumination for such testing 16. Notwithstanding this, it seems clear that dyers and printers will still have to employ accelerated fading tests using an artificial illuminant in their routine testing. The enclosed carbon are is widely used for this purpose; some means are required for indicating the amount of light-action to which a specimen has been exposed in the course of a test. One method is simply to state the duration of exposure, on the assumption that the intensity is known and constant. But the intensity is in general not known, and may vary considerably. Another method is to expose specimens of standard dyeings along with the test specimens, and to compare the amount of fading of the specimens and standards. This method also

element of the apparatus should fit into the specimen frame of the fading lamp along with the samples under test, and revolve in the frame with them. If the integrator is an electrical instrument, this would normally necessitate the use of slip-rings. What is required is a small, compact, completely self-contained unit which will fit into the specimen frame complete.

It has been found possible to construct an integrator to meet this requirement. The instrument is about the size of a small cigar box; it is a photoelectric light integrator, driven by a small hearing-aid battery, quite free from thermionic valves, using instead a small relay, and giving its reading on an internal telephone message register (Fig. 10 and 11).

#### REPEATING PATTERNS

Textile printers are concerned with repeating patterns; and these present several problems that are of interest from the scientific and mathematical points of view.



- Opal reflector
- Telephone r
- Battery
- Conden
- Guide pins
- Discharge resistor
- Photocell Moving-coll relay
- W Light regulator

Fig. 11-Compact Light Integrator for Fading Lamps (Interior View)



Fig. 12 - Determination of Outline Length of Colours in a Pattern

To cite a simple example, the cost of printing a given design depends to some extent upon what proportion of area is occupied by the printed colours. In principle, of course, this can be measured with a planimeter, but that method is much too slow for routine work, especially if the pattern contains much fine intricate detail. An approximate estimate can be obtained by laying a grid of lines across the design and measuring the intercept of any colour or colours on the grid lines, as a fraction of the total length of the lines. The possible accuracy of this method can be improved by increasing the number of lines. By scanning the design rapidly in a close helix as pictures are scanned for transmission by wire or radio, the accuracy can be still further increased without requiring too long for the determination.

A closely analogous problem of measurement exists in the case of the engraving of designs for textile printing. Nearly all engraving processes require hand work at some stage, and the amount of work required often varies according to the degree of intricacy of the design: in fact, it is proportional to the length of outline that has to be traced in drawing a unit of the repeating pattern. It is thus of interest to measure this length of outline. Again it is obviously possible to do this with a tracing wheel or opisometer, but the work is too slow and inconvenient to be practical. As in the case of the area measurement, it is possible to make the measurement by means of a grid of sampling lines. The method is to lay the sampling grid over the pattern unit, and to count the number of intersections made between the outlines of the colour areas and the grid lines (Fig. 12). Elementary theory shows that the total length of outline in the pattern unit is given by-

$$L = \frac{\pi}{9} \times C \times h$$

where C is the number of intersections and h is the distance between the sampling lines. This expression is strictly true only when the outlines of the pattern do not run predominantly in any single direction, but have an equal length of outline parallel to every direction. If the outlines are polarised parallel to some fixed direction, the error can rise to a maximum of 22% (for stripe patterns). The method would. of course, not be applied in the latter case; in the great majority of designs the error is not greater than 5%, and can be made considerably less by taking the average of counts on two grids at

right-angles. The possible accuracy is also increased by increasing the number of sampling lines. For four typical designs, the average probable error ranges from 13% for a single counting line to 1.3% for a hundred lines. It is obvious that in this case, also, the measurement could be mechanised by employing scanning methods.

#### APPLICATION OF OPTICAL INSTRUMENTS

Optical instruments have a considerable field of employment in textile printing. The Mutochrome (Hilger & Watts) is a commercial instrument which is a type of lantern projector which can be used to show in a very easy way how any given design would look in any possible colour combination.

Other optical instruments have been designed and constructed by finishing firms themselves. There are, for example, certain possible faults of drawing a design which are quite invisible in the unit of pattern, and become visible only when the units are assembled in repeat form as they are in the printed cloth. Such faults are the formation of objectionable diagonal stripes of light or dark, known as "runs". If they are discovered for the first time when the engraved pattern is printed or impressioned, considerable delay and expense may be incurred in re-engraving. To avoid this a form of optical projector has been constructed which projects an assembly of nine adjacent repeats of a given pattern unit-aligned, registered, and fitting together as they would fit when the design is engraved and printed-so as to permit inspection of the design from this point of view before engraving is commenced.

In the operation of a printing machine, there is a natural desire to run the machine as fast as possible. At high speeds of running, however, it becomes difficult if not impossible to see the printed pattern clearly to detect and correct any faults of register or other features. The use of a stroboscope is an obvious suggestion, but is not particularly helpful because the repetition frequency of the pattern is often much less than the sixteen or so per second that is required to give a steady comfortable picture. The situation is suitable for the employment of types of optical system with moving optical elements which are so synchronised with the movement of the cloth that they project a stationary picture. Appliances of this type have been constructed.

#### NON-TEXTILE MATERIALS IN TEXTILE PRINTING

In considering the application of scientific research to textile finishing, it is natural and proper to think first of the textile materials themselves, but it happens that in some sections of the art, in textile printing for example, certain other materials are of almost equal importance. Textile printers are directly interested in the consistency of their printing pastes, and in the metals of their printing rollers and cleaning doctors. Physicists in the printing industry are thus led to take an interest in problems of rheology, metallography, metallurgy, and surface finish.

The consistency of the printing pastes has been intensively studied by means of viscosity measure-

ments. It has been found that useful printing pastes possess a high viscosity at low rates of shear, falling to low values when the rate of shear is increased. The technical value of a printing paste depends upon the balance of these properties <sup>18</sup>. They also introduce some difficulties into the measurement, for there is no constant Newtonian viscosity that can be measured, and the rate of shear has to be accurately known. The problems involved have, however, been solved by physicists working in this field, and commercial instruments for routine work are now available. Some printing pastes may contain a residue of burst starch granules; this inhomogeneity makes microscopic examination also advisable.

The properties of the printing rollers that are of interest are the accuracy of form, the smoothness of surface, and the grain structure immediately underlying the surface. Accuracy of form can be examined by the ordinary methods of metrology, taking into account, however, that the copper may be a much softer metal than most to which metrological methods are applied.

### SURFACE SMOOTHNESS OF PRINTING ROLLERS

The smoothness of the roller surface is of considerable importance in printing, since, if the surface is rough, it may act like a shallow engraving, and thus carry and apply printing colour to areas of the cloth which should be free from it. In addition to this influence on "facing", surface

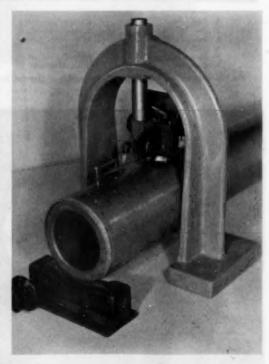
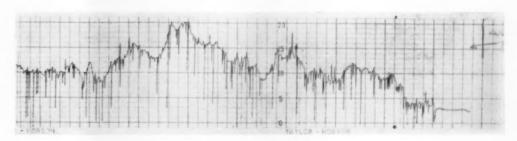


Fig. 17 — N.P.L. Tomlinson Surface Roughness Meter adapted for Roller Testing



 $\begin{array}{c} {\sf Magnificatioe-Vertical} \times {\sf 1000} \\ {\sf Horizoncal} \times {\sf 100} \\ \\ {\sf Fig.~13-Taylor-Hobson~Roughness~Trace~of~Machined~Roller~Surface} \end{array}$ 





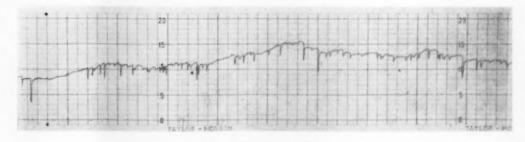
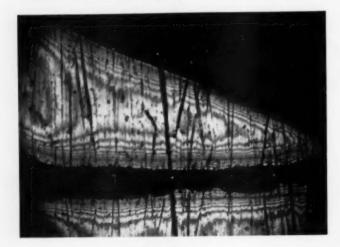
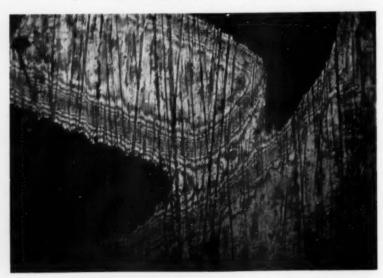




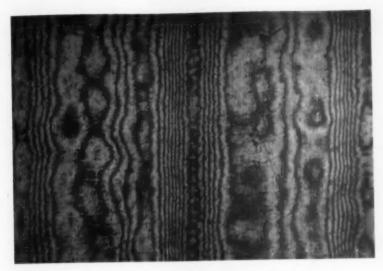
Fig. 18 - Interference Pattern at Contact of Roller Surface with Proof Plane



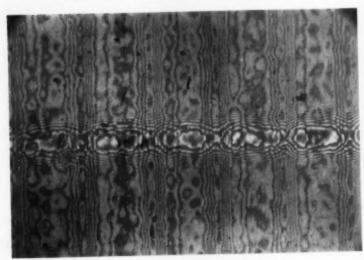
 $\label{eq:Fig. 10} \mbox{{\it Magnification}} \times 150 \\ \mbox{{\it EFig. 10}} \mbox{{\it Light-wave Interference Contour Map of Varnish Layer}$ 



 ${\color{red}{\bf Magnification}} \times {\bf 150}$  Fto, 20 — Light-wave Interference Contour Map of Varnish Layer



 ${\color{red}{\bf Magnification}} \times {\color{red}{\bf 40}}$  Fig. 21 — Light-wave Interference Contour Map of Varnish Layer



 ${\color{red}{\sf Magnification}} \times {\bf F6}$  Fig. 22 — Interpretation of Contour Fringes by Crossed Proof-plane Fringes

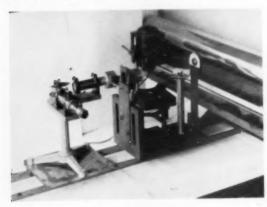
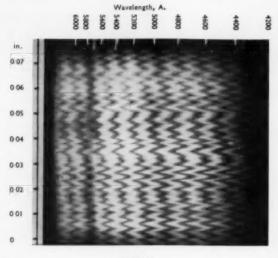


Fig. 23 — Apparatus for Testing Varnish Layer Thickness on Rollers by Spectrum Analysis of Reflected White Light



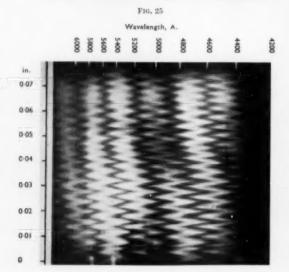


Fig. 24-26 — Varnish Layer Thickness Profile Fringes in Spectrum of Reflected White Light

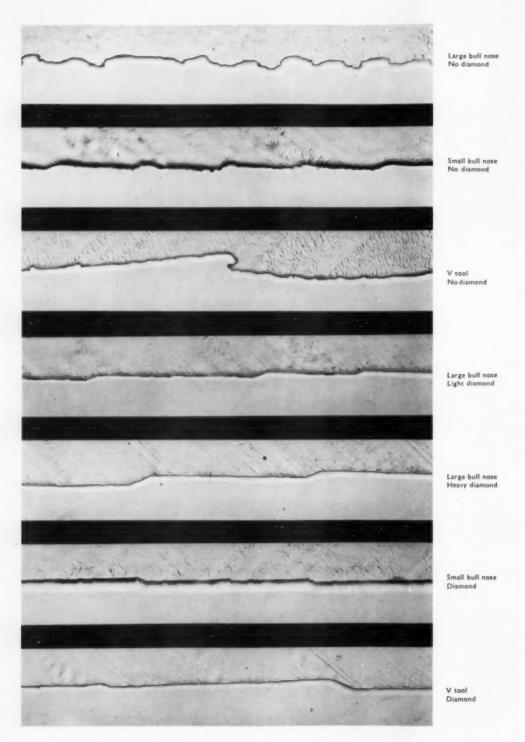
F16, 26



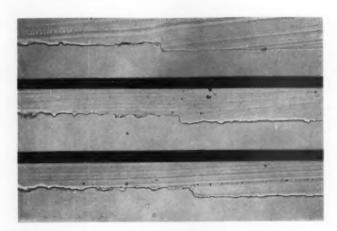
 ${\bf Magnification} \ \ \times {\bf 100}$  Fig. 27 — Transparent Replica and Cross-section of Turned Roller Surface



Magnification × 150 Ftg, 29— Photomicrograph of Transparent Replica of Defective Chromium Plating



 ${\color{red}{\bf Magnification}} \times {\color{blue}{\bf 500}}$  Fig. 28 — Cross-sections of Replicas of Machined Roller Surfaces



Magnification × 250

Fig. 30—Cross-sections of Replicas of Chromium Plating at Edge to show Thickness of Plating



Fig. 33



F1G. 34

 ${\bf Magnification} \ \times {\bf 50}$  Fig. 33 and 34—Cross-sections of Silvered and Coppered Engravings showing Grain Structure of Roller Copper

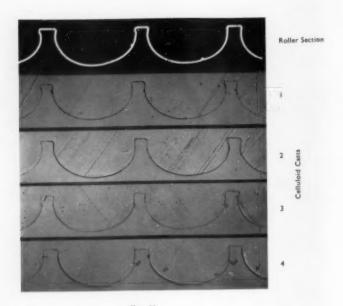
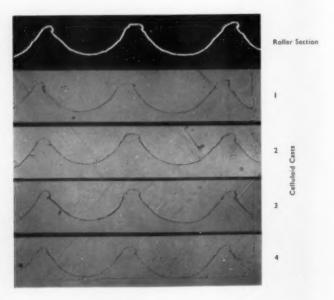


Fig. 31 and 32 — Cross-sections of Silvered Engraving Grooves compared with Cross-sections of Celluloid Casts of the Same Area



F10. 32

smoothness may be important in certain engraving

Studies of surface smoothness have been made by a variety of methods. Fig. 13-16 show profile traces of roller surfaces which were obtained on a commercial testing instrument. Fig. 17 shows a modified form of the Tomlinson instrument which was originally designed at the National Physical Laboratory. It is shown in use for testing a roller. Optical testing methods based on the observation of light-wave interference fringes, formed between the roller surface and a glass test-plate, have also been employed. Fig. 18 is a photograph under the microscope of the interference fringes formed in mercury green light at the line of contact between a roller surface and a flat plate of clear glass. The fringes form a contour map of the area, and the most central continuous fringes are a good profile of the surface, on a scale which can be determined.

In certain stages of the engraving process, films of mordant-resist varnish have to be formed on the surface of the roller. Light-wave interference is a useful method of measuring the thickness, uniformity, and local structure of the resist layer. Fig. 19 and 20 are photomicrographs of parts of such a resist, with an image formed in it. The photographs were taken by monochromatic light; interference fringes are formed within the resist layer itself, and each photograph displays an exact contour map of the layer thickness, which can thus be readily determined in every detail of variation.

Fig. 21 shows a photomicrograph of the contour fringes formed in the resist where there is no image. The fringes are quite clear, but in this case it is sometimes not easy to tell whether a given feature is a ridge or a valley. Fig. 22 shows how this point can be resolved by observing the cross-fringes which are formed when a transparent proof-plane is laid on the surface.

Another way of measuring the thickness of resist films on the rollers is to direct a beam of white light on to the surface, and to analyse the reflected light in a spectroscope. Fig. 23 illustrates the apparatus for measuring the film thickness on a roller by this method. The spectrum is crossed by dark bands, examples of which are illustrated in Fig. 24–26. The spacing of the bands depends on the thickness of the film, which can then readily be calculated from wavelength measurements. The waviness of the bands in the spectrum is a picture of the local variations of thickness of the resist.

#### FILM REPLICAS

In the study of roller surfaces, extensive use has been made of plastic replica methods. It is interesting to note that in recent years replica methods have been widely used for electron microscope work, but they were being used for optical microscopy many years prior to this. Direct observation of roller surfaces is sometimes awkward, but a transparent film replica of the surface can be very comfortably examined in the laboratory, using the best powers of a good microscope. For every feature of the surface that can be seen by reflected light, a corresponding feature can be seen when the film replica is

examined in transmitted light (Fig. 27). By suitable adjustment of lighting conditions, it becomes impossible to tell whether a given photograph is of a replica or of an original: the replica is a faithful copy down to the most minute detail. The replicas can be easily sectioned in a microtome, to show the profile of the surface (Fig. 28); if the surface is rough enough, the roughness can be measured in this way. In one case, for example, it was found that the colour capacity of the rough surface was about 1% of that of an average engraving. The roughness can also be studied in all its aspects by studying the optical refracting properties of the replica, in transmitted light.

Also in connection with "facing", if a roller which has faced is allowed to dry, and if a film replica of the surface is then made, the replica strips the entire colour film from the roller surface, and every feature of the density and the distribution of the facing colour can then be examined very conveniently in the replica.

The surface characteristics of chromium plating, which is often electrodeposited on rollers to increase their resistance to abrasion, can very readily be determined by the replica method. The photograph (Fig. 29) shows the formation of bubbles, craters, and cracks in the chromium layer. The thickness of chromium deposits can readily be measured by the height of the step in a cross-section of a replica made of the edge of a plated patch (Fig. 30). On a plated (or unplated) roller which has printed, the effects can be very clearly seen; they are, of course, chiefly due to the action of the printing-machine doctor blade.

#### MICROSCOPY

Microscopic examination plays a large and varied part in physical research on textile printing. A number of special techniques have been developed for this purpose, of which the film replica method may stand as an example.

A number of additional examples may be quoted from the study of the etched intaglio engravings which are used. In such engravings, the depth and the shape of cross-section of the grooves are of interest. These are often determined by making thick plastic replica casts of the engravings, in a material which is dimensionally stable, stripping the replicas when dry, cutting thin cross-sections in a microtome, and inspecting these under a microscope. The value of various plastic substances for this work has been studied by comparing cross-sections of their casts with crosssections of the engraving itself (Fig. 31 and 32). The latter sections were themselves cut by a technique in which the engraving has a thin layer of silver electrodeposited on it, followed by a thick layer of copper, so that the required profile is clearly visible but is embedded for sectioning in a massive block of uniform hardness. The sectioning is done by the usual metallographic methods, which incidentally reveal details of the crystal grain structure of the copper, right up to the surface (Fig. 33 and 34). The colour-carrying capacity of engravings can obviously be determined from the cross-sections, and, by means of colorimetric

measurements of the colour obtained on the face and the back of the printed cloth, can be related to the colour yield and degree of penetration, for example in experiments connected with the printing value of various thickenings.

In connection with the penetration of colour into cloth, problems of wetting also arise, so that measurements of surface tension and angles of contact have considerable significance. The same factors are important also in connection with the wetting of the roller surfaces by the printing colour. This is particularly evident in connection with chromium-plated rollers. A great deal of work has been done in the microscopy of textile fibres, for example to determine the location and state of particle division of colouring matter within them, and of other substances such as the synthetic resins which are applied in several present-day finishing processes. Most of this work involves the use of the microtome in cutting cross-sections of the fibres.

It would of course be possible to describe many more illustrations of the manifold applications of physics to the dyeing, printing, and finishing industries. It is not possible to cover the ground adequately in the course of a single paper; one can only describe a few instances which seem typical, and hope that they may also be interesting.

I would like, in closing, to call to mind once more the man in whose name these lectures are given, and the purpose for which, I think, they were founded.

John Mercer possessed the true scientific mentality. His North-country tenacity was a match for the "stubborn irreducible facts" which confronted him; his faith assured him beyond all question that the facts were understandable as instances of natural laws which were themselves grounded in an ultimate rationality, whose character would be disclosed in wider and wider generality, and in deeper harmony, by sustained enquiry. John Mercer would have got on quite well with the back-room boys.

In every generation the tide of scientific advance floods eagerly into the courses that have been presented to it by the labours of the preceding age. In our time the tide is flowing with formidable power, most strongly in the channel of physical science. But throughout, the adventure of science has been essentially the same continuing adventure; the physicist in industry today is exploring and building under the same ægis as that which sheltered and inspired John Mercer.

The writer's thanks are due to colleagues in the Research Department of the Calico Printers' Association Ltd., and particularly to Mr. G. S. Hibbert, the Head of the Department, for help, advice, and encouragement in the preparation of this paper. The writer also desires to thank the Chairman and Directors of the Association for permission to illustrate the theme of the paper by publishing certain details of work which has been done in the laboratories of the Association.

(MS. received 28th April 1954)

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#### Level Dyeing with Afterchrome Dyes

I. D. RATTEE

Meeting of the Huddersfield Section held at Field's Café, Huddersfield, on 15th April 1954, Mr. J. Calvert in the chair

The rate of dyeing of the unchromed afterchrome dyes has been determined under different dyeing conditions. It is considered that the sudden increase in exhaustion which occurs with many afterchrome dyes when formic or sulphuric acid is added to the bath towards the end of the dyeing to clean the bath prior to chroming is disadvantageous, and that the rate-of-dyeing properties of the dyes in dyebaths sufficiently acid from the commencement to effect satisfactory exhaustion are sufficiently good to achieve level absorption of dye. Also it has been found that the rate of levelling of the unchromed dyes in the presence of all the acid necessary to exhaust them is in general superior to those of the level-dyeing acid dyes. A method of classifying the level-dyeing properties of the unchromed afterchrome dyes has been worked out using two well known level-dyeing acid dyes as standards.

The properties of the afterchrome dyes during chroming and those of potassium dichromate itself have been examined. It has been found that the majority of the chrome dyes are chromed extremely rapidly. It has not been possible to confirm Gaunt's findings that dyes will preferentially migrate during chroming on to heavily chromed parts of the wool. It has been found that the alkali formed due to reduction of dichromate has an effect sufficiently powerful to cause desorption of absorbed ions, and it is considered that uneven chrome absorption may cause uneven desorption, rather than uneven absorption, of dye ions. Some recommendations are made for improving the chroming stage of dyeing.

#### Introduction

A considerable amount of experimental work is described in the literature on the dyeing properties of the level-dyeing acid dyes which has been of great practical value to the dyer. However, in the field of chrome dyeing the greatest amount of research has been carried out on the theoretical aspects of chromate dyeing, when in fact it is the afterchrome process which is used to by far the greatest extent in dyeing with chrome dyes. This trend in research has to some extent been reversed, as evidenced by the recent work of Lister 1 and Gaunt <sup>2</sup>, and this paper represents a further contribution to this field of work.

Although the chromate dyeing process offers considerable advantages over the afterchrome process from the point of view of time of dyeing, the afterchrome process possesses a number of outstanding advantages, which more than offset its longer duration. Of the three methods for applying chrome dyes—chrome mordant, chromate, and afterchrome— it is only by the last process that full advantage may be taken of the excellent level-dyeing properties of the unchromed chrome dyes. The dye may be applied in a similar way to the level-dyeing acid dyes and then converted very simply into the chrome lake, which has high wet fastness. The afterchrome process also possesses the advantages of giving dyed wool in better condition, higher dyebath exhaustion, and relative cheapness <sup>2</sup>. Thus the afterchrome process is preeminent in the production of heavy, dull dyeings of high wet fastness on wool.

In the main the afterchrome process is used for the dyeing of slubbing and loose wool. The reasons for this appear to reside in the lack of detailed knowledge of the level-dyeing properties of the afterchrome dyes. However, the increased interest in the application of fast colours to yarn arising from the introduction of the S.D.C. No. 2 wash test, although to some extent satisfied by the development of the Carbolan Salt A process, has aroused increased interest in the possibilities of applying chrome dyes to yarns by the afterchrome process for the production of heavy dull shades,

and at the present time quite considerable amounts of yarn are being dyed afterchrome. This trend, of course, adds to the need for a more detailed knowledge of the level-dyeing properties of the afterchrome dyes, such knowledge being at the same time of great interest to the slubbing and loose-

wool dyeing trade.

It has been shown 3 that the production of level results with level-dyeing acid dyes is influenced by two factors-initial strike and levelling in the boiling dyebath. The application of chrome dyes by the afterchrome process adds to these two a third stage, namely chroming. It is certain that in the first two stages, the afterchrome dye behaves qualitatively in a similar manner to the leveldyeing acid dyes, and the same conditions for the production of satisfactorily level results obtain. The third stage, chroming, is rather more complex in that dichromate must be absorbed by the wool and then reduced, and the chrome lake of the dye must be formed on the fibre. Two important factors exist here, namely even absorption and even reduction of dichromate. Although some work has been done on these two factors 4, not a great deal of information is available with regard to the effect of the second factor with different dyes.

The investigation of the afterchrome dyeing process described here has been made on the basis of the three stages referred to above, and is described under them for the sake of clarity.

#### Experimental

THE RATE OF DYEING OF AFTERCHROME DYES

The method of determination of the rate of dyeing of the afterchrome dyes was to set a dyebath with 1% of the dye, 3% acetic acid (30%), and 10% Glauber's salt in a 40:1 liquor ratio, all in respect of 21 g. of botany serge.

40 c.c. of the dye liquor was removed as standard. Twenty 1-g. pieces of botany serge which had previously been wetted out in boiling water were entered into the dyebath set at 20°c., and the temperature was raised by 2°c. per minute. At each 10°c, temperature interval one 1-g, piece was removed along with 40 e.c. of the dye liquor. This procedure was carried out until the boil was reached, after which the time interval between taking samples was increased to 15 min. After 45 min. boiling, 1% formic acid (85%) (based on the 9 g. of wool remaining in the bath) was added, and samples were taken after 5, 10, and 15 min. further boiling. The degree of exhaustion was determined by comparison of optical densities in the Spekker absorptiometer. A graph was plotted of exhaustion against time and temperature.

The experiment was repeated using a dyebath set with 3% acetic acid (30%), 1% formic acid (85%), 10% Glauber's salt, and 1% of the dye, taking samples at 10°c. temperature intervals while raising the temperature by 2°c. per minute up to the boil.

....

TABLE I
Time of Half-dyeing of the Chrome Dyes

Colour Index			Dyeing A	Method-B
No.			(min.)	(min.)
proper.	Solochrome Yellow 3G		2.2	1.9
36	Solochrome Yellow WN		4-1	2.5
-	Solochrome Yellow 2GN		6.5	4-4
-	Solochrome Yellow 2G		> 85	22.8
343	Solochrome Yellow C		11.8	8.3
	Solochrome Yellow L		26-5	12.8
197	Solochrome Yellow Y		29.6	17-2
	Solochrome Flavine R		> 85	15.8
219	Solochrome Flavine G		17.5	5.5
-	Solochromate Fast Orange	2R	15.4	5.1
274	Solochrome Orange GR		18-6	12.5
-	Solochrome Brown EB		36-0	29-6
101	Solochrome Brown MG		2.3	2.3
	Solochromate Fast Brown 3	G	9.6	7.6
104	Solochrome Brown MO		11.0	11.0
101	Solochrome Brown M		12.0	7.8
-	Solochrome Brown RG		16.9	13.3
98	Solochrome Brown RH		17.0	9.3
****	Solochrome Brown P		28.0	23.7
-	Solochrome Brown R		28.2	21.0
-	Solochromate Fast Red G		16.0	13.0
652	Solochrome Red ER		19-3	13-5
214	Solochrome Red D		22.2	17-6
-	Solochrome Brilliant Red 31	3	39.0	21.0
216	Solochrome Red B		42.0	17-7
-	Solochromate Fast Violet B		10.3	8-5
169	Solochrome Violet R		18-5	15-5
proces	Solochrome Fast Violet RL		20.6	10.8
722	Solochrome Cyanine R		7.0	19-7
202	Solochrome Dark Blue B		14.5	12.2
720	Soloehrome Azurine B		16.3	10-1
-	Solochromate Fast Blue B		24-0	22.0
-	Solochrome Fast Navy 2R		25-0	16.0
292	Solochrome Green V		27.5	23.9
1085	Solway Blue Black B		23.0	16-7
parent.	Solochromate Fast Grey B		16.0	13.5
201	Solochrome Black 6BN		20.5	18-2
-	Solochrome Black PG		21.8	19-4
299	Solochrome Black F		22.6	22.6
204	Solochrome Black A		24-5	23.4
203	Solochrome Black WDFA		30.5	26-9
170	Solochrome Black PV		14.5	10.7

A-3% Acetic acid (30%) + 10% Glauber's salt exhausted with 1% formic acid (85%).

B-3% Acetic acid (30%) + 1% formic acid (85%) + 10% Glauber's salt.

From the rate-of-dyeing graphs, the time required to reach 50% exhaustion was determined. This time was termed the time of half-dyeing (50%), and the values for the different dyes examined by the two dyeing methods are shown in Table I.

All the dyeing experiments were carried out in dye beakers heated in a paraffin bath. The rate of rise of temperature was controlled by a Simmerstat. On reaching the boil, the beakers were covered by a round-bottom flask containing cold water in order to minimise evaporation losses.

The results also indicate the effect of the addition of the exhausting acid on the dyebath exhaustion. In Table II, the dyebath exhaustion values just prior to the addition of the exhausting acid and at the end of the dyeing are shown together with the dyebath exhaustion where all the acid is added at the commencement. It will be observed that, with the dyes Solochrome Yellow 3G and Solochrome Yellow 2GN, an exhaustion of under 50% is achieved just prior to adding exhausting acid. This is due to the fact that with these dyes it was found that maximum exhaustions of 77% and 72% were obtained in each respective case after about 20 min. boiling and that dye was then desorbed to give the exhaustion value obtained in Table II. Where no figure is given, the result was not determined.

The increased exhaustion that occurred when the exhausting acid was added was found in all cases to take place within 5 min. of the addition, i.e. in many cases very rapidly.

The effect of increasing the acetic acid addition with a view to avoiding the necessity of using formic acid was examined with additions of up to 6% acetic acid (30%) on the weight of material. Insufficient exhaustion was obtained in most cases.

The use of 3% formic acid (85%) was found to give adequate exhaustion, but the rate of dyeing was very rapid. Glauber's salt additions of up to 25% had insufficient effect to give any improvement over the use of acetic and formic acids mixed.

## THE RATE OF LEVELLING OF THE CHROME DYES

The levelling properties of individual chrome dyes were determined on a semi-quantitative basis as follows—8 g. of botany serge was dyed to 1% depth in a 50:1 liquor with the dye under examination, 3% acetic acid (30%), and 10% Glauber's salt. The dyeing was commenced at 20°c., raised to the boil over 30 min., and boiled for 45 min. The bath was exhausted by adding 1% formic acid (85%) and boiling for a further 15 min. A "blank" dyeing, i.e. as above but without dye, was carried out on 8 g. of botany serge at the same time. At the end of the dyeing time, cuttings were taken from the two pieces, the liquors from the two dyebaths mixed, and the two patterns boiled together in the mixed liquor. Cuttings were taken from the patterns after 15, 30, 45, 60, 90, and 120 min. further boiling.

TABLE II

#### Effect of Addition of Exhausting Acid on Dyebath Exhaustion

		Dyebath E	xhaustic	n (%)—
Colour		Before	At End	When
Index		adding	of	all Acid
No.		Exhausting Acid	Dyeing	added at Start
	Solochrome Yellow 3G	43-0		56-2
36	Solochrome Yellow W2		80-3	67-6
90	Solochrome Yellow 2G		63-5	63-8
	Solochrome Yellow 2G	44-7	89-0	66-4
343	Solochrome Yellow C.	95.3	99.0	
343			00.0	98-5
197	Solochrome Yellow L .		89-3	84-8
197		65-1	95.7	98-9
010	Solochrome Flavine R	42.2	66-7	83.4
219	Solochrome Flavine G	56-5	82.0	83.5
-	Solochromate Fast	01.5		08.1
274	Orange 2R	91.7	00 =	97-1
274	Solochrome Orange GR		90-5	94-8
101	Solochrome Brown EB		00.0	73-6
101	Solochrome Brown MG	85-8	90-8	87.5
	Solochromate Fast	<b>80.</b> 6		00.0
104	Brown 3G	78-6		86-0
104	Solochrome Brown MC		79-2	60-4
101	Solochrome Brown M	86-6	92.3	73.9
-	Solochrome Brown RG		95.9	97.2
98	Solochrome Brown RE		73-1	73-2
and the same of	Solochrome Brown P .		-	87-3
Married .	Solochrome Brown R	64.5	-	80.7
-	Solochromate Fast			
	Red G	87-0	91.3	89-2
652	Solochrome Red ER .	90-0	95-6	93-4
214	Solochrome Red D .	82-6	88-4	84-2
-	Solochrome Brilliant			
		56-5	77-1	77-3
216	Solochrome Red B .	66-4	96-1	92.5
-	Solochrome Fast Violet B	92-6		97-9
169				0.0
109	Solochromate Violet R	72.8	77-5	83-0
-	Solochromate Fast Violet RL	51.4	66-4	67-8
722	Solochrome Cyanine R		00.4	83-3
202	Solochrome Cyanine R	74-6		93.3
202	Blue B	90-8		95.0
720	Solochrome Azurine B	86-7		93-8
-	Solochromate Fast	00.1		000
	Blue B	97-0	-	97-1
-	Solochrome Fast			
	Navy 2R	83-6	-	91-9
292	Solochrome Green V .	100	-	100
1085	Solway Blue Black B	74-6	_	92-1
	Solochromate Fast			
	Grey B	83-8	percen	91-4
201	Solochrome Black 6BN	89-0	-	94.9
porto	Solochrome Black PG	77-6	*****	88-0
299	Solochrome Black F .	93-9		100
204	Solochrome Black A .	94-0	-	96-8
203	Solochrome Black			
	WDFA	83-9	and the same of	87-8
170	Solochrome Black PV	80-4	90-0	86-4

The control dyeings at 0.25% and 0.75% depth of the test dye were prepared by the same dyeing method. They were equivalent to the position at half-levelness, and by comparison of these two patterns with the results of the levelling test it was possible to estimate the approximate time taken to achieve half-levelness. In this way it was possible to estimate the values for the chrome dyes with the exceptions of Solochrome Browns MO

TABLE III

#### Approximate Half-levelling Times of the Chrome Dyes

TIME	OF HALF-LEVELLING < 15 min.
	Solochrome Red D
	Solochrome Brown RH
	Solochrome Yellow 2GN
	Solochrome Yellow 3G
	Solochrome Brilliant Red 3B

TIME	OF HALF-LEVELLING 15-30 min.
	Solochrome Black PV
	Solochrome Yellow 2G
	Solochrome Yellow WN
	Solochrome Flavine G
	Solochrome Flavine R
	Solochromate Fast Violet RL

TIME	OF HALF-LEVELLING 30-45 min.
	Solochromate Fast Red G
	Solochromate Fast Grey B
	Solochrome Cyanine R
	Solochrome Violet R
	Salachrama Orange CR

TIME	OF HALF-LEVELLING 45-60 min.
	Solochrome Azurine B
	Solochrome Brilliant Violet BR
	Solochrome Fast Navy 2R
	Solochromate Fast Orange 2R

Time of Half-levelling 60-90 min.
Solway Blue Black B
Solochrome Red B
Solochrome Brown MO

Time of Half-levelling 90-120 min.
Solochromate Fast Brown 3G
Solochrome Yellow Y
Solochrome Brown RG

TIME	OF HALF-LEVELLING > 120 min.
	Solochrome Brown P
	Solochrome Brown R
	Solochrome Brown EB
	Solochrome Dark Blue B
	Solochrome Green V
	Solochrome Red ER
	Solochrome Yellow C
	Solochrome Black A
	Solochrome Black 6BN
	Solochrome Black F
	Solochrome Black PG
	Solochromate Fast Violet B
	Solochromate Fast Blue B

and M. In these cases marked deterioration of the dye due to prolonged boiling under acid conditions prevented assessment. The results are shown in Table III.

THE PROPERTIES OF CHROME DYES DURING CHROMING, AND THE PROPERTIES OF POTASSIUM DICHROMATE

#### (1) Rate of Absorption of Dichromate during Chroming

It is well known that both potassium dichromate and potassium chromate obey Beer's law very well up to quite high optical density values. Thus the exhaustion of a chroming bath may be determined from Spekker measurements as with dyes. This property was used for the determination of the

rate of absorption and reduction of dichromate in

the following experiments. A blank dyebath (1.4 litres) was set with 3% acetic acid (30%), 1% formic acid (85%), and 10% Glauber's salt based on 28 g. of wool. Fourteen 2-g. pieces of botany serge were wetted out in boiling water and immersed in the dye liquor at The temperature was raised to the boil over 30 min. and the dyebath was boiled for 45 min. The wool was lifted and 1% potassium dichromate on the weight of wool was added. A sample of the liquor was taken for standard purposes. The wool was re-entered and boiling continued for 5 min. After this time, a 2-g. pattern was removed, together with 100 c.c. of liquor. The pattern was hydroextracted, weighed, and air-dried. The 100 c.c. of liquor was basified by the addition of 0.1 g. caustic soda. This procedure was repeated after 10, 15, 20, 25, 30, 35, and 40 min. boiling. The exhaustion of the dichromate was determined by comparison of the optical density values of the samples with that of the original standard. Each of the 2-g. patterns was extracted in 70 c.c. of pH 8 buffer for 24 hr. by the method of Race, Rowe, Speakman, and Vickerstaff 4. Following extraction, the liquor was decanted, and the pieces were squeezed (retaining the liquor), washed twice with 10 c.c. of distilled water, and squeezed again. The total extract was made up to 100 c.c., and the optical density values were compared with those of standard chromate solutions. The chromium which had been extracted in this way represented the unreduced portion of the chromium absorbed. Correction was made for the chromium carried out mechanically as liquor when the pieces were removed from the bath. From these results it was possible to calculate the percentage of the total dichromate which had been exhausted and reduced at any time. The results are shown in Table IV.

TABLE IV

Rate of Absorption and Reduction of Dichromate in the Afterchroming Bath

Die	Reduction			
Time of Boiling (min.)	Exhaustion of Dichromate (%)	Dichromate on the Fibre (%)	Total Dichromate present (%)	
5	69-8	71-1	49-0	
10	75-5	85.9	64-2	
15	82.6	89-9	74-3	
20	89-4	91.7	81-9	
25	92-2	94-4	87-1	
30	95-5	93-4	89-1	
35	97-2	98-6	95-9	
40	98-8	98-5	97-4	

The determination of the rate of absorption of chromium by wool using optical density values introduces some inaccuracies owing to the presence of small quantities of reduced chromate in the liquor. The colour of these salts would, of course, be in the green region rather than the yellow, and it was considered that the errors were too small seriously to affect the results obtained.

#### (2) Rate of Absorption of Dichromate from Different Dyebaths

Four blank dyebaths were prepared each of 450 c.c. containing—

- (a) 3% Metachrome mordant
- (b) 3% Acetic acid (30%), 10% Glauber's salt, and 1% potassium dichromate
- (e) 3% Acetic acid (30%), 1% formic acid (85%), 10% Glauber's salt, and 1% potassium dichromate
- (d) 3% Sulphurie acid (98%), 10% Glauber's salt, and 1% potassium dichromate.

All percentages in the above refer to 9 g. of wool. The temperature of the baths was adjusted to 20°c., and a 50-c.c. sample removed as standard from each bath. To each bath sixteen 0.5-g. pieces of wetted-out botany serge were added, and the temperature was raised by 2°c. per minute.

TABLE V

Rate of Exhaustion of Chromium from Different Acid Dyebaths

Time	Temp.		Dyebath			
(min.)	(°c.)	(a)	(b)	(c)	(d)	
5	30	-	7-7	41.8	48-8	
10	40	_	19.5	-		
15	50	5-4	38-2	49.0	56-7	
20	60	_	34.0	_	-	
25	70	4-8	26.5	40.8	56.7	
30	80	4.5	22-8	45-4	46-4	
35	90	4.8	26.2	45-7	49-3	
40	100	2.1	27.8	45-7	70-4	
55	100	21.4	42.5	75.0	86-6	
70	100	47.6	81.5	86.5	100.0	
85	100	64.9	4-100	-	-	

Samples (25 c.c. of liquor and one 0.5-g. piece) were removed at each  $10^{\circ}$ c. temperature interval up to the boil and every 15 min. after reaching the boil for up to 1 hr. of boiling. The percentage exhaustion of chromium was determined from optical density comparison as before. The results are shown in Table V.

#### (3) Rate of Chromation of Different Chrome Dyes

The rate of development of the fibre–chrome–dye complex has been determined qualitatively as follows—16 g. of botany serge was dyed with 1% of the dye, 3% acetic acid (30%), and 10% Glauber's salt in the normal manner, and the dyebath exhausted with 1% formic acid (85%). A cutting was taken from the dyeing and 1% potassium dichromate added. Boiling was continued for 1 hr., during which cuttings were taken at intervals of 10, 20, 30, and 60 min. after adding the dichromate. From the results an estimate was made of the time taken to achieve full colour development. The results are shown in Table VI.

#### (4) Migration Properties of Chrome Dyes during Afterchroming

There are a number of references in the literature to the development of unlevelness by afterchrome dyes during the afterchroming stage <sup>4</sup>. It has been suggested that this is due to migration of dye preferentially on to areas where the concentration of reduced chromium is higher. The question of which dyes actually exhibit or are likely to exhibit this phenomenon was examined as follows—Botany serge (4 g.) was dyed to 1% depth with a

chrome dye by the normal method up to the At the same time blank afterchroming stage. dyeings were carried out on each of two 1-g. patterns of untreated botany serge and botany serge which had been sweet-chromed so as to contain 2.83 mg. Cr<sub>2</sub>O<sub>3</sub> per gram of wool. At the end of the dyeing time, the three dye liquors were mixed, and the three pieces, from which small sample pieces were first removed, were entered into the boiling mixed liquor. 1% Potassium dichromate (based on the 8 g. of wool present) was added, and boiling continued for 30 min. The pieces were then removed, dried, and examined for the degree of transference of dye to the originally undyed pieces and also for differences in the degree of transfer to the prechromed and untreated originally undyed pieces.

#### TABLE VI

#### Time required for Full Colour Development of Chrome Dyes in Chroming

CHROMING TIME 10 min. Solochrome Yellow C Solochrome Yellow 2GN Solochrome Orange GR Solochromate Fast Orange 2R Solochrome Brown RG Solochrome Brown R Solochrome Brown P Solochrome Brown EB Solochromate Fast Brown 3G Solochrome Red D Solochromate Fast Red G Solochrome Violet R Solochromate Fast Violet B Soloehrome Brilliant Violet BR Solochrome Azurine B Solochrome Cyanine R Solochrome Dark Blue B Solochromate Fast Blue B Solway Blue Black B Solochrome Fast Navy 2R Solochrome Green V Soloehrome Black F Solochrome Black PG

CHROMING TIME 20 min.
Solochrome Yellow 3G
Solochrome Yellow 2G
Solochrome Red B
Solochrome Red ER
Solochrome Brilliant Red 3B
Solochrome Black A
Solochrome Black 6BN
Solochrome Black PV

CHROMING TIME 30 min.
Solochrome Yellow WN
Solochrome Flavine G
Solochrome Bravine R
Solochrome Brown MG
Solochromate Fast Violet RL

CHROMING TIME 60 min. Solochrome Brown M Solochrome Brown MO Solochrome Brown RH The experiment was repeated for a large number of chrome dyes, and those exhibiting appreciable migration during afterchroming are listed in Table VII. In no case was more migration on to prechromed wool observed. Where there were differences in depth of colour between prechromed and normal wool, these could all be accounted for by the initial difference in colour between the two wools.

#### TABLE VII

#### Migration Properties of Chrome Dyes during Afterchroming

Solochrome Yellow 3G
Solochrome Yellow 2G
Solochrome Yellow 2GN
Solochrome Yellow WN
Solochrome Flavine G
Solochrome Brilliant Red 3B
Solochromate Fast Violet®RL

Solochrome Flavine R
Solochrome Orange GR
Solochrome Red D
Solochrome Brown RH
Solochrome Violet R
Solochrome Brilliant Violet BR
Solochrome Azurine B
Solochrome Cyanine R

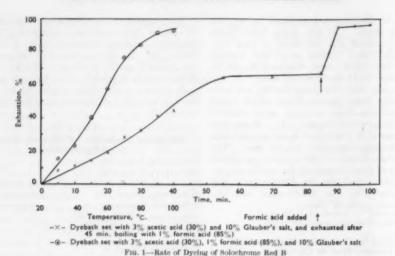
#### Discussion of Results

The data on rate of dyeing shown in Table I demonstrate that the chrome dyes as a class have a fairly low rate of dyeing under the conditions examined. It has not been possible to obtain even a general relationship between rate of dyeing and constitution as with the level-dyeing acid dyes 3. However, an important feature of any comparison between the chrome and acid dyes is the fact that under their preferred dyeing conditions 85% of the level-dyeing acid dyes have a time of half-dyeing of less than 9-5 min., whereas this applies to only 12% of the chrome dyes examined. Thus from an acetic acid dyebath the chrome dyes, as a class, are absorbed considerably more slowly than are the level-dyeing acid dyes from a sulphuric acid dyebath.

The main weakness of the otherwise excellent dyeing properties of the chrome dyes in this respect is the very marked increase in rate of exhaustion which occurs with a number of the dyes when the exhausting acid is added, prior to chroming. From Table II it can be seen that almost half of the dyes exhibit this marked increase in exhaustion with addition of exhausting acid.

However, if the exhausting acid is added at the commencement of dyeing, this sudden exhaustion of the dyebath does not occur, the same final exhaustion is achieved, and the rate-of-dyeing properties of the dyes still compare favourably with those of the level-dyeing acid dyes. This is best illustrated by the rate-of-dyeing graph of Solochrome Red B shown in Fig. 1.

If level-dyeing acid dyes are taken which have known even strike, viz. Lissamine Fast Red B and Disulphine Blue AN (C.I. 673), which have halfdyeing times of 4-0 and 13-5 min. respectively



determined under their preferred dyeing conditions, it can be shown that only 10% of the chrome dyes examined dye more rapidly than Lissamine Fast Red B and only 50% dye more rapidly than Disulphine Blue AN, even when all the acid is added at the commencement. Thus it may be concluded that all the acid necessary to procure satisfactory exhaustion may be added at the commencement of dyeing, that this procedure will bring the advantage of avoiding the sudden exhaustion of part of the dye just prior to chroming, and that, while the rate-of-dyeing properties of the dyes are worsened by this method, they remain much superior to those of the level-dyeing acid dyes.

The second important property of the dyes is that of levelling. If the results of Table III are assessed against the two level-dyeing acid dyes Lissamine Fast Red B and Disulphire Blue AN with half-levelling times of 30–45 min. and 60–90 min. respectively, determined under their preferred dyeing conditions and by the same method as described above, it can be seen that the levelling properties of the chrome dyes applied from a dyebath, containing all the necessary acid, are very good. 40% of the dyes possess levelling properties at least as good as those of Lissamine Fast Red B and nearly 60% at least as good as those of Disulphine Blue AN.

Included in those dyes which level at least as well as Lissamine Fast Red B are almost all the chrome yellows and Solochromes Brown RH, Red D, Brilliant Red 3B, Cyanine R, and Violet R, all of which are regarded in practice as level-dyeing types. On the other hand, Solochromes Fast Navy 2R and Red B and Solway Blue Black B, which also are regarded as level dyeing, level only at a rate intermediate between those of the two standards. Clearly, with these dyes factors other than levelling condition their general dyeing behaviour.

The level-dyeing properties of dyes are a composite of their rate-of-dyeing and rate-of-levelling properties. With dyes of the level-dyeing acid type, dyeing is very often so rapid that their rate-of-dyeing properties may be disregarded as a controllable factor, and their level-dyeing properties assessed on the basis of their levelling properties alone. The unchromed chrome dyes dye considerably more slowly than most of the level-dyeing acid dyes, and in any assessment of level-dyeing properties it is necessary to take both rate-of-dyeing and rate-of-levelling properties into account.

From the results of Tables I and III it is possible to classify the level-dyeing properties of the unchromed dyes applied from a dyebath containing 3% acetic acid (30%), 1% formic acid (85%), and 10% Glauber's salt on the weight of goods, using the dyeing behaviour of Lissamine Fast Red B and Disulphine Blue AN as standards of reference. The dyes may be classified into five groups—

- 1. Very level dyeing
- 2. Level dyeing
- 3. Moderately level dyeing
- 4. Not level dyeing
- 5. Very unlevel dyeing

The dyes may be put into these groups in accordance with the following scheme—

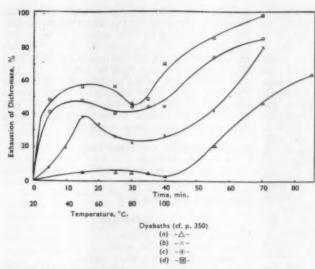
Group 1 includes dyes which dye more slowly than Disulphine Blue AN and level more rapidly than Lissamine Fast Red B.

GROUP 2 includes dyes which-

- (a) dye at a rate intermediate between those of the two standards and level more rapidly than Lissamine Fast Red B; and
- (b) dye more slowly than Disulphine Blue AN and level at a rate intermediate between the two standards.

GROUP 3 includes dyes which-

- (a) dye and level more rapidly than Lissamine Fast Red B; and
- (b) dye and level at rates intermediate between those of the two standards.



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Group 4 includes dyes which dye and level more slowly than Disulphine Blue AN.

GROUP 5 includes dyes which dye more rapidly than Lissamine Fast Red B and level more slowly than Disulphine Blue AN.

More emphasis has been placed on levelling properties than on rate-of-dyeing properties in this system of classification. This has been done because, although rate of dyeing is more important with the chrome than with the level-dyeing acid dyes, it is almost certain that levelling is still the more important factor. The result of the classification with the dyes examined is shown in Table VIII.

#### TABLE VIII

#### Level-dyeing Properties of Chrome Dyes

GROUP I (VERY LEVEL DYEING)
Solochrome Flavine R
Solochrome Yellow 2G
Solochrome Violet R
Solochrome Cyanine R

Solochromate Fast Grey B GROUP 2 (LEVEL DYEING)

Solochrome Flavine G Solochrome Brown RH
Solochrome Orange GR Solochromate Fast Violet RL
Solochrome Red B Solochrome Fast Navy 2R
Solochromate Fast Red G Solway Blue Black B
Solochrome Black PV

GROUP 3 (MODERATELY LEVEL DYEING)

Solochrome Yellow 2GN Solochromate Fast Orange 2R Solochrome Yellow 3G Solochrome Brown MO Solochrome Yellow WN Solochrome Azurine B

GROUF 4 (NOT LEVEL DYEING)

Solochrome Yellow Y
Solochrome Brown P
Solochrome Black A
Solochrome Black F
Solochrome Black PG
Solochrome Red ER
Solochrome Black GBN
Solochromate Fast Blue B
Solochrome Black WDFA

GROUP 5 (VERY UNLEVEL DYEING)
Solochrome Yellow C Solochromate Fast Brown 3G
Solochromate Fast Violet B Solochrome Dark Blue B

This classification refers to the level-dyeing properties of the dyes when all the acid is present at the commencement of dyeing. Nevertheless, it fits in extremely well with the known properties of dyes based on practical experience. The value of such a classification, this being the case, is that it provides a method whereby a new dye or an unfamiliar dye may be quickly assessed, and many of the less desirable features of learning by experience may be avoided. Two apparent anomalies appear in the classification. Solochrome Red ER and Brown EB fall into Group 4. This is due to their inferior levelling properties. They do, however, possess very good rateof-dyeing properties and are normally dyed in full maroons or browns, where their levelling properties are of less importance. Thus in the depths at which they are normally used these dyes have gained a

deserved reputation for level dyeing, although it is possible that they are not suitable for yarn dyeing. The classification cannot take such special considerations into account, and gives an assessment of the level-dyeing properties of the dyes at all depths. Another instance of this is the fact that almost all the chrome blacks are described as not level dyeing. This is perfectly correct when these dyes are considered over all depths, although in practice unlevel results in blacks are very rare because of the weight of dye applied. The classification cannot be used, therefore, without a certain amount of discretion in relation to such questions.

Solochrome Brown RH represents a third anomaly. This dye, although possessing excellent level-dyeing properties prior to chroming and being correctly placed in the classification from that point of view, possesses a marked tendency to develop unlevelness during afterchroming. Careful

chroming, however, can avoid this difficulty.

What the classification does show is that there is a satisfactory range of chrome dyes (Groups 1 and 2) possessing excellent or good level-dyeing properties, from which may be selected dyes for the dyeing of yarns, paler shades, etc., where levelness is somewhat of a problem.

The results also cast some light on the problem of uneven dyeings developing during the chroming stage. The results in Table IV show that dichromate is reduced by the wool at the boil very rapidly indeed. Table VI shows that the chrome lake is developed also at a very rapid rate, and no uneven migration has been detected when chroming is carried out under conditions designed to give uneven reduction. It may be concluded from these results that the development of uneven dyeings during chroming owing to migration in the normal sense does not occur. There is some interesting indirect evidence for an alternative cause of this phenomenon in the results of Table V. For

clarity these are shown graphically in Fig. 2. It can be seen that under all conditions of chroming dichromate is absorbed steadily to a greater or lesser extent up to 60-70°c., and that it is then desorbed and reabsorbed. The most reasonable explanation that can be given of this phenomenon is that in the temperature region 60-70°c, reduction commences with the liberation of caustic potash in the fibre, thereby raising the internal pH and causing desorption of dichromate ions. If this can occur with dichromate ions, it is reasonable to suppose that it can occur also with dye anions. Thus it is possible to visualise a situation in which dichromate ions, having been absorbed unevenly, are reduced to give varying internal pH values in different parts of the load. This can lead to desorption of dye to different extents and the production of uneven dyeings.

This theory, of course, does not differ from the previously expressed view that unevenness in chroming is due to migration on to heavily chromed parts of the wool, in so far as the implied solution to the problem is concerned. In either case the answer to the problem appears to lie in the procure-

ment of even initial dichromate absorption in the first place. From the data obtained the commencement of chroming at 70°c. appears to be recommended. At this temperature, reduction has already commenced, but the rate of uptake is slow. The temperature may then be raised to the boil over 20 min., and boiling continued for 10–20 min. The very rapid rate of chromation of the dyes at the boil enables full colour development, any unchromed dye present on reaching the boil being fully chromed under these conditions.

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(MS. received 29th March 1954)

#### References

- 1 Lister, J.S.D.C., 65, 97 (1949).
- <sup>1</sup> Gaunt, ibid., 70, 46 (Feb. 1954).
- <sup>8</sup> Lemin and Rattee, ibid., 65, 217 (1949).
- <sup>4</sup> Race, Rowe, Speakman, and Vickerstaff, *ibid.*, **54**, 141 (1938).

#### CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

#### The Solubility of Wool in Urea-Bisulphite Solution and its Use as a Measure of Fibre Modification

The alkali solubility test <sup>1,2</sup> has been extensively used as a means of arbitrarily measuring chemical changes in wool brought about in such chemical processes as chlorination, peroxide bleaching, carbonising, and acid dyeing. The solubility of untreated wool under the controlled conditions normally employed in the test is about 11-14%, and increases when the wool is processed to an extent which depends on the conditions of the treatment, viz. temperature, time, concentration of reagent, etc. The increase is probably caused by hydrolysis of the main peptide chains or fission of the disulphide linkages or both.

Many of the processes to which wool is subjected are carried out under alkaline conditions, e.g. scouring, crabbing, setting, and decatising. In the alkali solubility test, alkali-treated wool gives a lower result than untreated wool because either some part of the alkali-soluble protein is removed in the alkaline treatment itself or the formation of new resistant cross-linkages in the wool causes its actual solubility to decrease. The decrease is necessarily small and, as a measure of the effect of an alkaline treatment, the alkali solubility test as at present used is insensitive.

It appeared possible that the effect of alkaline treatment on the solubility of wool would be more clearly revealed by the use of a more powerful protein-dispersing agent than  $0.1 \, \text{M}$ , sodium hydroxide as used in the alkali solubility test. Jones and Mecham<sup>3</sup> have shown that the keratin

proteins can be partly dispersed in a neutral solution of a protein denaturant, e.g. urea, in the presence of a reducing agent, e.g. sodium bisulphite, capable of splitting the disulphide linkages, an essential preliminary reaction in the dispersion of the keratins. Hence a treatment which totally or partly replaces the disulphide by linkages resistant to reduction would be expected to decrease the solubility of the protein in such a reagent and thus to enable small changes to be detected. Horn, Jones, and Ringel 4, by isolating lanthionine from alkali-treated wool, have shown that it contains sulphide linkages, which are resistant to the action of reducing agents.

			TABLE I		
Treati	ment		Solubility in Urea- Bisulphite (% on dry wt.)	Cystine Sulphur Content (% on dry wt.)	Alkali Solubility (% on dry wt.)
*Untreated		***	47-3	2-67	-
30 min. in 0 at 30°c.			33.7	2.68	_
30 min. in 0 at 50°C.	·ln·Na	CO <sub>3</sub>	25-2	2.59	-
30 min. in 0 at 70°C.		CO,	2.5	2.37	
*Untreated	***		52-6	2.95	13-4
Boiled for 3	hr. at				
pH 8-2	***		8-1	2.36	13-5
Boiled for 3	hr. at				
pH 2·2			72-4	2.91	35-0

The samples of untreated wool referred to were undyed worsted yarn and were not identical.

It has now been shown that, when wool is treated in alkali, its solubility in urea-bisulphite solutions decreases, and it can be rendered virtually completely insoluble by a moderately severe alkaline treatment. On the other hand, treatment of the wool under acidic conditions causes the solubility to increase as in the alkali solubility test.

The effects of different treatments on the amount of wool dissolved in 1 hr. by a solution containing 50% urea and 3% sodium bisulphite at 65°c. are given in Table I.

Thus the method shows promise as a means of indicating the nature of the treatment which a sample of wool may have received, and is also a sensitive method of assessing the severity of such treatment.

As an example of the practical use of the test, samples taken from two pieces of the same quality of undyed cloth differed in respect of handle and colour, one (B) being much yellower than the other. The results (Table II) obtained on the pile yarn suggest that the handle and the colour of sample B had been impaired by excessive treatment in alkali.

TABLE	II	

B Cystine sulphur, % 2-18 2.03 Urea-bisulphite solubility, % ... 41-9

The method is being actively investigated for a wider range of processes, and the results will be K. LEES F. F. ELSWORTH reported later.

WOOL INDUSTRIES RESEARCH ASSOCIATION "TORRIDON" LEEDS 6

10th May 1954

- <sup>1</sup> Harris and Smith, Bur. Stand. J. Res., 17, 577 (1936).
- <sup>2</sup> Lees and Elsworth, J.S.D.C., 68, 207 (1952).
- <sup>2</sup> Jones and Mecham, Arch. Biochem., 3, 193 (1943). <sup>4</sup> Horn, Jones, and Ringel, J. Biol. Chem., 138, 141 (1941).

#### Solid Dyeings on Wool-Terylene Mixtures

I read with great interest the recent article in the Journal on Problems of the Garment Dyer and Finisher, including the Dyeing of Buttons 1. I have the impression that many of our members feel that too few of this essentially practical kind of

paper appear, and the authors are to be congratulated on producing such an excellent example of this type of work.

I should, however, like to make some observations on the paragraphs dealing with the dyeing of wool-Terylene mixtures, because the authors, by implying that the behaviour of Cibacet Orange 4R, in invariably dyeing the wool more deeply than the Terylene, is typical of disperse dyes, may have given too discouraging an impression of the possibilities open to dyers concerned with this problem. Disperse dyes vary considerably in their affinity for animal fibres, and amongst available dyes having good affinity for Terylene, Serisol Yellow 2G, Serisol Brilliant Blue G, and Serilene Pink BT have been found to give the minimum staining of wool. It appears from laboratory trials that, in the dyeing of pale shades on Terylene-wool mixtures, the stain produced on the wool by these three dyes is so slight that they may be applied along with acid dyes in a single bath to give satisfactory dyeings.

With heavy shades, of course, heavier staining of the wool occurs, although at 95-100°c. with these selected dyes, the Terylene is much more deeply dyed than the wool if the two fibres are present in similar proportions in the mixed fabric. Even so, because of the poor fastness properties of disperse dyes on wool, it is advisable, after first applying the disperse dye, to clear the stain from the wool as completely as possible before finally dyeing the wool with acid dyes from a fresh bath. The clearing treatment, carried out in a bath containing soap or a dispersing agent such as Dyapol PT. also helps to remove any relatively loose surface colour from the Terylene as well as any "carrier" such as diphenyl which may have been employed.

Thus, whilst it carrot be claimed that the dyeing of this mixture of fibres is at all a simple operation, there is no doubt that correct choice of dyes is an important factor in achieving a satisfactory solution.

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<sup>1</sup> Kramrisch and Bratt, J.S.D.C., 70, 173 (May 1954).

## Notes

#### Meetings of Council and Committees July

Council-2nd and 14th

Publications -- 20th

Colour Index Editorial Panel - 27th

Diplomas-7th

Diplomas Executive Subcommittee-7th and

Fastness Tests Co-ordinating-20th

#### A.S.D.C. Lectures in Manchester

To assist prospective candidates for the Associateship diploma, especially in preparing for papers A and B, the Textile Chemistry Department of the College of Technology will offer a special course during the session beginning October 1954. In planning the course, it will be assumed that those wishing to take part have already passed the final examination for the A.M.C.T. in Textile Chemistry or have reached an equivalent standard. The course may also interest others who do not propose to take the examination.

The classes will be held on two evenings a week, and will consist of lectures on theoretical aspects of colour chemistry, the general chemistry of high polymers, modern theories of dyeing processes, and colour theory. Those on colour theory will be accompanied by laboratory work. It would help in the organisation of the course if those who are interested would write to the Department of Textile Chemistry, College of Technology, Manchester I, who will supply more detailed information as soon as it is available.

#### Lectures on Sizing

A series of six post-advanced lectures on sizing will be held at the Royal Technical College, Salford, on Mondays (7–9 p.m.) beginning on 8th November 1954. Full details may be obtained from the Honorary Secretary, Manchester and District Advisory Council for Further Education, Education Offices, Deansgate, Manchester 3.

#### H. E. Fierz-David (1882-1953)

Prof. H. E. Fierz-David graduated at the Zürich Polytechnic, then studied under Sir M. O. Forster at the Royal College of Science, London, and, after varied industrial experience with the Brunner Monel Nickel Co. in South Wales, at a brewery in Munich, and for nine years with the Geigy Co. in Basle, returned to the Zürich Polytechnic as Professor of Technical Organic Chemistry, where he remained from 1917 to 1952. His researches there were mainly in the field of Colour Chemistry and Dyeing, but he was interested also in topics such as the cause of rancidity in fats and the structure of viscose and acetate rayon.

In the chemistry of intermediates, he studied the catalytic chlorination of nitrobenzene and nitrotoluene, catalytic reductions with hydrogen in aqueous solution, the preparations of aniline-osulphonic acid, monoethyl-o-toluidine, and resorcinol from the alkali melt of benzene-m-disulphonic acid, sulphonation in the naphthalene and anthracene series, and the sulphuration of ethylbenzylaniline.

Among dyes, the azo class chiefly engaged his attention, but his publications were mainly of theoretical interest and concerned constitution and reactivity; e.g. Fierz-David established that the p-hydroxyazo dyes are in the quinonoid form in neutral or acid solution, whereas in alkaline solution a hybrid azo-hydrazone structure exists. He also prepared 2:3-phenylazonaphthol, which was orange yellow in colour and quite different from its red 1:2 and 1:4 isomers, its spectrum being similar to that of 2-phenylazonaphthalene, and its hydroxyl group not functioning as an auxochrome. In the sulphur dye field, Fierz-David did a large amount of pioneering work, and established the constitutions of Pyrogen Indigo, Hydron Blue R, Immedial Pure Blue, and Pyrogen Green.

In the indigo field his 6:6'-dichloroindigo and green-blue 2:3:2':3'-naphthindigo failed to find commercial use, but in artists' colours he discovered that cobalt and nickel borates produced very beautiful bright fast shades. The constitution of the Turkey Red lake was found to be alizarin (4 mol.), Al (2 atoms), Ca (3 atoms), and water of crystallisation (5 mol.), but if crystallised from pyridine the lake contained pyridine (2 mol.) and water of crystallisation (7 instead of 5 mol.); the Turkey Red oil employed in the dyeing did not become incorporated in the lake.

Fierz-David will also be gratefully remembered for his valuable treatises on colour and allied chemistry, such as Grundlegende Operationen der Farbenchemie, Künstliche organische Farbstoffe, and Die Entwicklungsgeschichte der Chemie.

(Abstracted from *Helv. Chim. Acta*, **37**, 427-435 (March 1954)).

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#### OBITUARY NOTICE

#### Frederic Leathley Goodall

A PERSONAL APPRECIATION

By the death of Frederic Leathley Goodall, the colour manufacturing and using industries are left significantly the poorer. To the members of the Society of Dyers and Colourists the loss is great, for not only have we lost a most eminent member, but most of us have lost also a valued friend. His death at the comparatively early age of 52 came as a great shock to all, and especially to those who saw him, so recently, actively carrying out the onerous duties of President.

F. L. Goodall was educated at Leeds Grammar School and the Department of Colour Chemistry of Leeds University, where he graduated B.Sc. with First Class Honours in 1922. He obtained the M.Sc. degree the following year. During his time at the University he was quiet and reserved, but he enjoyed fencing, winning the championship and representing the University in the fencing team. He played rugby football for the Old Leodiensians for some seasons, becoming Secretary and Vicecaptain.

By his studies of the theory and the practice of wool dyeing he rendered great services to the wool dyeing section of the textile industry, which were recognised by his election as a Fellow of the Royal Institute of Chemistry in 1944 and by the award of the Gold Medal of the Society in 1940.

In later years he found relaxation in fishing, golfing, and reading. He became a very keen fisherman, and such was his modesty that he never boasted of his achievements nor sought to magnify them. He was also a very ardent Churchman, and will be long remembered for this. In spite of his heavy responsibilities in business, he was always

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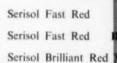






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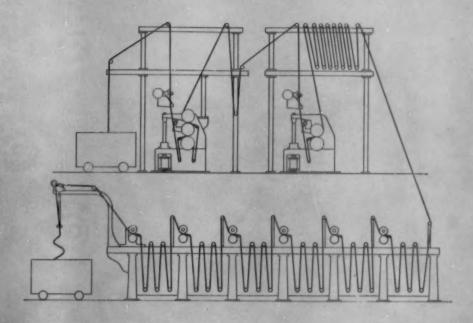
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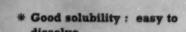
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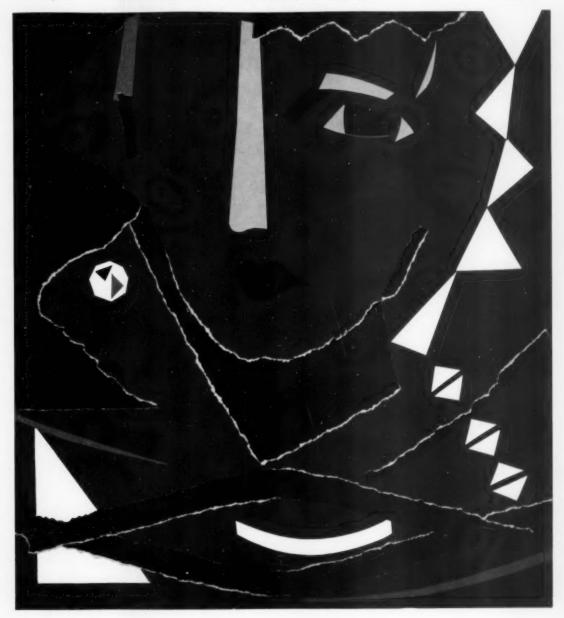
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cheerful and courteous, and his capabilities as an organiser and a chairman were obvious to all who had the pleasure of serving with him in the Society.

His record of service to the Society, which he joined as a Junior Member in 1920, is a striking testimony to his interest in it and the dyeing industry. He was a member of Council for sixteen years and a Vice-president from 1950 to 1953. At various times he served on many committees, including the Finance Committee (1932–1949), Charter Committee (as Convener), Publications Committee (1929–1949), Fastness Tests Committee, West Riding Section Committee, and more recently the Diplomas Committee. He represented the Society on several external bodies, notably the International Federation of Textile Chemists and Colourists. He was made a Fellow of the Society in April of this year.

He looked on his election as President of the Society in 1953 as a very great honour, and graced this office with efficiency and charm. In this he was very ably assisted by his wife, Marie. His work for Junior Members will be long remembered, and his enthusiasm for the Diplomas Scheme did much to bring this to fruition. He was disappointed at the failure of the Society to obtain a Charter, and had he lived would have worked with enthusiasm to bring about this desirable result.

He is survived by his wife and two daughters. We shall all miss Eric Goodall very greatly.

A. BREARE

#### A COLLEAGUE'S APPRECIATION

F. L. Goodall was a Yorkshireman—a fact of which he was very proud—being a native of Leeds and receiving his education at Leeds Grammar School and at Leeds University. On graduation in 1922 he was awarded a Clothworkers' Research Scholarship. For the next year he worked in collaboration with the late Professor A. G. Perkin, and with him published a paper on "Some Reduction Products of Hydroxyanthraquinones".

In 1923 he accepted a position as chemist with C. R. Roberts & Co. Ltd., who were then the Geigy agents for Yorkshire. Later, on the formation of the Geigy Colour Co. Ltd., he became chief chemist, and subsequently was appointed Yorkshire Area Manager.

In 1948 he migrated to Manchester to become Sales Director of the Geigy Co. Ltd., and was nominated Joint Managing Director in March 1953. Other firms on which F. L. Goodall served as Director were Geigy (Holdings) Ltd., Ashburton Chemical Works Ltd., and Gyl Chemicals Ltd.

In all these capacities, in the words of the Chairman of the Geigy Company, "he diligently applied his great knowledge of the tinctorial arts, and also inspired all those responsible to him with his leadership".

On behalf of the Company he visited Australia and America, and particularly in the latter country his technical abilities and wide knowledge of dyeing were appreciated in many quarters.

No reference to F. L. Goodall would be complete without placing on record the high esteem in which he was held throughout the Geigy organisation in Basle, his knowledge and experience, especially in the wool field, being recognised and fully appreciated. His excellent knowledge of German was but one weapon in his armoury which proved of great value in his international contacts.

Outside his family life and his business contacts, his main interest had been the Society of Dyers and Colourists, of which he had been a member since 1920, when he joined the Leeds Junior Branch. We in Geigy regarded his election to the Presidency as a natural and fitting climax to many years of devoted service. During the brief period in which he held office he looked upon his position as one to be fulfilled at all costs, and those of us who knew him intimately realised that on many occasions these responsibilities were upheld in spite of difficulties experienced through ill-health.

His interest in technical education is shown by his membership of the Textile Industries and Dyeing Committee of Leeds University and the Chemistry Sectional Committee of Manchester College of Technology.

To us in Geigy he was known affectionately as "F.L.G.". In his business career he functioned in many rôles—scientist, technician, and administrator—but that in which we shall remember him best is one which he filled with charm and understanding—the rôle of "guide, philosopher, and friend". To say that he will be missed is something of an understatement, but we as a Company and as individuals are deeply conscious of the gap which his departure has created in our ranks.

J. HOLT

#### FUNERAL

The funeral service was conducted by the Rev. E. D. Reeman (*Rector of Wilmstow*) in the chapel of the Lawnswood Crematorium, Leeds, on Wednesday, 30th June 1954, at 12.45 p.m.

Among family mourners were Mrs. F. L. Goodall, Miss P. M. Goodall and Miss H. J. Goodall, Mr. and Mrs. D. M. Goodall, Mr. and Mrs. E. A. Hallas, Mr. A. C. Hallas, Mr. and Mrs. W. Clough, Mr. and Mrs. A. Leathley, Miss B. Leathley, and Dr. and Mrs. W. L. Nicholson.

The Society was represented by Dr. C. J. T. Cronshaw, Mr. F. Scholefield (also representing the Association of British Chemical Manufacturers), and Dr. C. M. Whittaker (Past Presidents); Mr. Fred Smith (President-elect); Mr. C. O. Clark and Mr. H. Foster (Vice-presidents); Mr. A. Breare, Mr. H. Hampson, and Mr. J. V. Summersgill (Ordinary Members of Council); Mr. D. B. F. McAndrew (representing the Scottish Section), Mr. S. R. Meadows (Chairman of the Midlands Section), and Mr. G. S. J. White (Chairman of the Manchester Section); Mr. H. Jennison (Honorary Treasurer), Mr. J. G. Hopkinson (Honorary Secretary), and Mr. J. Barritt (Chairman of the Publications Committee; also representing Mr. A. R. Baines, Chairman of the Wool Textile Research Council); and Dr. C. J. W. Hooper (Editor) and Mr. J. W. Nicholls (Secretary).

The Geigy Company Ltd. was represented by Mr. G. A. Campbell, Mr. C. A. Geigy, Mr. H. Jones, and Mr. B. H. Walker (Directors); and Mr. G. L. Thornton, Mr. J. Holt, Mr. C. R. Wilkinsson, Mr. L. M. Hesse, Mr. J. Schofield (also representing Mr. E. Isles), Mr. G. Whitehead, Mr. H. Wood, Miss B. M. Sunderland (also representing Mr. W. Poole), Miss J. P. Winpenny, Mr. R. Grico, Mr. A. R. Mason, Mr. R. Turton, Mr. G. P. Emmett, Mr. C. Hobday, Mr. F. Rath, Mr. W. Beal, Mr. E. Musgrave, Mr. G.

Clilverd, Mr. J. Heaton, Mr. J. Watson, Mr. J. Murry, and Mr. J. Jacques.

Geigy (Holdings) Ltd. was represented by Mrs. G. Parkes (representing Mr. G. Parkes), Mr. and Mrs. C. F. Gysin, Mr. H. Clayton, and Mr. H. L. Addleshaw; Ashburton Chemical Works Ltd. by Mr. A. Smith, Mr. H. Stromberg, Mr. G. A. Tillotson, and Mr. B. J. Warr; Geigy Pharmaceutical Laboratories by Mr. E. Asher, Mr. R. Kenyon, and Mr. H. Worthington; and James Anderson (Colours) Ltd. by Mr. P. Whitaker (representing Mr. A. H. Whitaker).

Among other organisations, the Textile Institute was represented by Mr. R. J. Smith (Honorary Treasurer), Mr. J. Boulton (Honorary Secretary), and Mr. D. B. Moore (General Secretary); the University of Leeds by Prof. W. Bradley, Prof. J. B. Speakman, and Mr. F. Manchester; Bradford Technical College by Dr. R. L. Elliott; the Dyers' and Finishers' Association by Mr. W. R. Mathers (Chairman); the Board of Trade (Dyestuffs Control) by Mr. C. G. Hulse; Bradford Textile Society by Mr. G. G. Hopkinson; and the Bradford Liberal Club by Mr. S. M. Haigh, Mr. W. D. Knight, and Mr. M. Wilkinson.

Among other friends and business associates present at the funeral were Mr. P. F. Crosland, the Rev. E. N. Pedley (Vicar of Burley-in-Wharfedale), Mr. and Mrs. A. E. Battle, Mr. G. Yashpon, Mr. J. H. Nuttall, Mr. H. Jowett, Mr. R. Heaton, Mr. A. L. Wood, Mr. R. Hullah, and Mr. F. M. Stevenson; Mr. E. M. Padgett (representing Mr. J. Ewing) and Mr. E. Wilson; Mr. D. E. Jowett; Mr. R. B. W. Muirhead; Mr. R. B. Whitohead; Mr. H. Jackson and Mr. H. Crook; Mr. W. P. Walker, Mr. N. Ellse, Mr. P. Christen, and Mr. E. Shrive; Mr. F. A. Helme, Mr. D. G. Marshall, and Mr. H. Wiles; Mr. B. Diggle (also representing Mr. R. J. Hannay); Mr. T. Harrington and Mr. G. K. Day; Mr. J. Moorhouse, Mr. H. Swift, and Mr. N. Stephenson; Mr. M. G. White; Mr. J. E. Mossey; Mr. F. Harrison; Mr. E. Sunderland; and Mr. M. Swithenbank.

#### New Books and Publications

#### **Der Colorist**

#### Eine Monographie des Textilchemikers

By R. Haller. 2nd edition 1953. Pp. 117. Heidelberg: Verlag Melliand Textilberichte. Price, DM 8.80.

It is remarkable, and perhaps regrettable, that the term Colourist, which figures so prominently in the name of our Society, is to be found so rarely in any other connection on the pages of our Journal. Some of our members who are not closely associated with the art and science of calico printing may think that the term is merely a convenient general expression to cover the activities of those engaged in the coloration of materials by any process (not necessarily dyeing); this view, however, scarcely does justice to the term in its specific application in the calico-printing trade, where the colouristor "chemist-colourist"- not only concerns himself with many operations and phenomena that are chemical, physical, or mechanical in nature (some only remotely related to the actual process of coloration), but also works on a higher colouristic plane than the dyer-just as the dyer works on a higher plane than the bleacher. The bleacher, it is true, works very precisely and cleanly within the limited field of colours that may be described commercially as white, and the dyer (who may also be a bleacher) matches colours with great precision over the whole gamut; but the print-works colourist (who may be a bleacher, and is certainly a dyer) works with another dimension-colour harmony-and his appreciation of this property in a multicolour print is of greater significance than his ability to produce a match for a given colour.

The interested reader who wishes to know more of the functions of the colourist (particularly the would-be colourist in search of advice on his prospective career and the training that it demands) should turn to Prof. Haller's booklet, now available in a second, enlarged edition; but if he is not familiar with the present state of the industry, particularly in this country, he must remember that Haller's description refers primarily to the Continental colourist of the first quarter of the century, the somewhat false impression given by the book in its first edition (1939) being only partly remedied by the inclusion in the second of an additional section on changes in the status of the colourist (the booklet should perhaps be renamed "Der alte Colorist"— the title of a jocular ditty that is published as an appendix). Haller points out, the manufacturers of dyes and auxiliaries have largely assumed the function of working out new printing processes and developing methods of applying their products, and the book will have served a useful purpose if it prompts a few of those who might otherwise become slaves to the pattern card to recognise that there is still scope for ingenuity and resourcefulness. It must be remembered also that there is an increasing tendency for division of labour and delegation of duties to occur—thus in some organisations the crucial judgments on hue and colour harmony

may be made by non-technologists. It is doubtful, also, whether colourists are able, under the conditions now generally prevailing (particularly in commission printing), to exert any great influence on public taste and so succeed in their "cultural mission", which is the subject of another additional

section (9 pages).

Apart from these and a few other additions, the original text is reprinted practically without change, and the remarks made in Mr. F. Scholefield's review of the first edition (J.S.D.C., 56, 514 (Dec. 1940)) still apply and are fully endorsed by the present reviewer. The accumulated experience of so great a colourist as Prof. Haller (who recently celebrated his eightieth birthday\*) is not to be taken lightly, and the book is commended not only to young or prospective colourists, but also to the old stagers, who cannot fail to find much of interest and value, even if they cannot always agree with the views expressed.

The book is bound in board covered with tastefully printed cotton material and is frontis-

pieced with a portrait of the author.

A. E. STUBBS

\*We regret to report that Prof. Haller died on 23rd July 1954.

The Structure of Textile Fibres
An Introductory Study

Edited by A. R. Urquhart and F. O. Howitt, Pp. 165. Manchester: The Textile Institute

1953. Price, 12s. 6d.

Eminent scientists occasionally unbend, and when they do some of them write books of popular science. Sometimes the more frivolous works of eminent men have outshone or outlasted their serious works, as in other fields has happened to Sullivan and Dodgson. Popular science has a long and honourable tradition, as anyone will testify who has enjoyed reading the lighter works of Boys, Bragg, or, in our own field, Astbury. This is a book of popular science, or perhaps we should say of popular technology. It is a conscious and praiseworthy attempt to interpret for laymen modern ideas on fibre structure.

In making an assessment of the book the principal question to answer is how successfully does it perform its task of interpretation? This is a question of language in the sense in which Willard Gibbs said that mathematics was also a language. One can make no progress unless one understands the meaning of the symbols. In examining the language here, the reviewer has tried to imagine how a reader would react who came fresh to the matter in the text. Looked at like this, the pace at which chemical and physical concepts are introduced suggests that it is suitable for laymen conversant with chemistry and physics up to matriculation level.

Thirteen authors have collaborated to produce as many chapters. General concepts and detailed examples are given. The text ranges over the chemistry, physics, morphology, and mechanical properties of fibres and relates these to structure and constitution.

With everyone so busy nowadays and with the rapid pace at which new knowledge is accruing, the most convenient way to produce a new book is by the collaboration of a number of authors as here. Yet this procedure has its special difficulties, the chief of which is that of obtaining uniformity of treatment, no matter how hard the editors may work. For example, here Clibbens and Lord illustrate the structure of cotton with a straight photomicrograph, whereas Howitt illustrates wool and Turner flax with different kinds of schematic drawings. One feels that it would have helped the tyro had it been possible to use the same conventions for all.

The contents of the book appeared first as a series of articles in the *Proceedings of the Textile Institute*. They have been reprinted in revised form here. The book will be a convenient text for students and technologists at an intermediate stage of their education, and will provide a helpful introduction to more advanced works.

J. M. PRESTON

#### Handbuch der chemischen Untersuchung der Textilfaserstoffe

Erster Band

Vorarbeiten und allgemeine Methoden der Feuchtigkeitsbestimmung Qualitative und quantitative Faseranalyse Anhang—Röntgenographische Faseruntersuchung

By H. M. Ulrich. Pp. xviii + 330. Vienna Springer-Verlag, 1954. Price, DM 54.00.

Although the general title of these new analytical texts mentions only chemistry, the contents of this first volume also includes physical tests of fibrous materials.

The first section is a miscellany of tests and occupies one-eighth of the text. The second section, which is of similar length, is concerned solely with the moisture contents of fibres. Next comes a section, about half the book, on the identification of fibres. After this is a section on their quantitative analysis, which completes the text except for an appendix on X-ray methods.

There is a most detailed list of contents at the beginning and a decimal guide at the end, but there is no index. Several textile analytical texts have appeared without indexes, but this is no reason for continuing a bad practice. As it is, the reader who wishes to refer to a subject either by its description or by the name of the author has to search twelve pages of contents and may still miss his aim.

This is a reference book, not a text for reading. Its main sections are on the analysis of fibres, and cover much the same ground as in Luniak's book (cf. J.S.D.C., 65, 453 (1949) and 70, 189 (May 1954) ). The treatment is generally very similar and indeed some of the matter has been borrowed from him. but the new text is more comprehensive and less selective. There is a large and useful collection of literature references. Because of the absence of an index, one cannot make the test of looking up a random selection of subjects to see whether they are included and how they are treated, but browsing through the text shows that the classical tests are fully described. The newer chromatographic methods and those based on infrared absorption are not considered, whilst the tests based on refractive indices and X-rays receive comparatively

scant attention. There are a few photomicrographs and drawings of fibres.

The book should be available in libraries and can be recommended for reference purposes.

J. M. Preston

#### Starch and its Derivatives

By J. A. Radley. 3rd edition 1953. Volume I: Pp. xi + 510; Volume II: Pp. xi + 465. London: Chapman & Hall Ltd. Price, 65s. 0d. each volume.

All textile chemists are familiar with the second edition of Mr. Radley's book, published in 1943, for they have found it to be a reliable source of information. They will therefore welcome the appearance in two volumes of a revised and enlarged third edition. The general method of presentation adopted in the second edition has been maintained, but additional chapters have been included and others have been completely rewritten and brought up to date. The author has had the assistance of many recognised authorities in the field of starch chemistry, and their contributions give added significance to the work. The first volume is divided into two parts, one dealing with the structure and reactions of starch and the other with amylases and their action on starch. Of the seventeen chapters in Part I, eleven are contributed by specialists and they include a discussion on the biological function of starch by Dr. S. Peat and an account of the chemical evidence for the structure of starch by Dr. L. Hough and Dr. J. N. K. Jones. Professor E. F. Degering of the Buckman Laboratories Inc. has contributed chapters on the esters, ethers, and inorganic derivatives of starch as well as one on the oxidation of starch, while other authors have discussed topics such as starch retrogradation, starch and the hydrogen bond, the waxy cereals and starches which stain iodine red, and dextrin and dextrinisation. The chapter on the starch fractions has been written by Dr. T. J. Schoch, who has included some of his own original work. Part II will be particularly useful to those concerned with desizing, for here are described in detail the preparation of enzymes used in the starch industry and the action of  $\alpha$ - and  $\beta$ -amylases on starch. An account is also included of methods of determining the activity of amylase preparations.

Volume II is concerned with more industrial topics, and is divided into three parts. In Part I the manufacture of starch and starch products is discussed, chapters on wheat, rice, sago and corn starches, glucose and maltose, dextrin and British gum, and physically modified starches being included, as well as one on the production of chemicals from starch by fermentation processes. Part II, on the industrial applications of starch, is necessarily somewhat sketchy, for it covers an extremely wide range of topics-adhesives, the paper industry, the textile industry, the rôle of starch in bread staling, the food industry, and the utilisation of the by-products of starch manufacture. The chapter concerning textiles deals mainly with sizing and desizing and the use of starch in printing. It will be useful mainly to

chemists and others requiring a general survey, but workers in the textile industry will find it rather disappointing. This may not be entirely the fault of the author, for considering the importance of sizing and desizing in the textile industry, the published literature on these topics is particularly uninspiring. Textile chemists will appreciate Part III, for it is an account of methods of examining and analysing starch and starch products, and they will find the collection of photomicrographs of various starches extremely useful for reference.

The main feature of the books is the ample documentation and the very large number of references, which include papers published before and during 1950. These are comprehensive and complete and are conveniently classified. The work is well produced, and although more expensive than its predecessor it should be purchased by all who have need of up-to-date and authoritative information on starch. It will become a standard work, and the author and his collaborators are to be congratulated on the successful completion of the extremely difficult and laborious task of presenting the vast literature on starch in a readable and systematic form.

C. S. WHEWELL

#### A Text-book of Macro and Semimicro Qualitative Inorganic Analysis

By Arthur I. Vogel. 4th edition 1954. Pp. xviii + 663. London: Longmans, Green & Co. Price, 22s. 0d.

On first opening this book, one is immediately impressed by the clarity and the attractiveness of the format in which the revised text has been reset. Further, the chapter number and the section number appropriate to the text are given as page headings, so that cross-references can be quickly located.

In Chapter I, in which the theoretical basis of qualitative analysis is discussed, the most important addition is an account of the Brønsted–Lowry acid–base theory, together with a discussion of hydrolysis based on the theory. Chapter II deals with the experimental techniques of macro-semimicro-, and micro-analysis using apparatus which, apart from the spectroscope and the centrifuge, is simple and inexpensive. Micro-analysis here simply means the scaling down to milligram quantities, still using traditional schemes of separation.

The reactions of cations, including dropreactions, are discussed in Chapter III, where also are to be found tables for separating cations on the macro- and semimicro-scale. Alternative Group II tables are provided for the use of either ammonium sulphide or sodium hydroxide in the separation of the arsenic group. Chapter IV discusses the reactions and some separations of anions; tests using the newer reagents are included.

The next two chapters, V and VI, offer, respectively, a systematic scheme of macro-analysis for elementary students and a systematic introduction to micro-analysis. Both schemes use the tables for cation separation given in Chapter III.

For advanced students, Chapters VII and VIII provide a scheme of macro-analysis which is almost completely self-contained, for the author has not hesitated to repeat textual and tabular material given previously. The reactions of the rarer elements are given in Chapter IX together with tables for the identification of one or two of them in presence of the commoner cations.

The final chapter deals with the technique and some applications of paper chromatography to inorganic separations. The elegance of these methods will captivate most students and may even prompt impish queries as to whether much of the book is now really necessary!

Teachers and students who are conversant with the previous edition will realise that many improvements have been made during the revision and that the rearrangement made has clarified the underlying division into elementary, semimicro, advanced, and "rare element" schemes of analysis. The author has retained the traditional system of group precipitations and separations and uses hydrogen sulphide for Group II cations. Not everyone may agree with this. The student will, however, learn something of the art of inorganic analysis, and, equally important, may have his only opportunity (apart from quantitative work) of becoming acquainted with some of the properties of inorganic compounds.

The statement on p. 5 that, in the electrolysis of aqueous sodium chloride, the liberated sodium atom reacts with water to produce hydrogen is rather old-fashioned, and should be amended to conform to the more acceptable explanation based on the hydrogen overpotential at the cathode.

Perhaps in a future revision the author will include something about hydrazine and hydroxylamine, both of inorganic lineage, but seldom found in any textbook on analysis.

This new edition will greatly increase the reputation of this valuable textbook, which is well suited for students at any level up to Honours Degree standard.

A. B. CRAWFORD

## Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes,
Any publication abstracted may be referred to by members of the Society on application to
Dr. C. B. Stevens, Design Department, Leeds University

#### Clayton Dyestuffs Co. Ltd.

Chlorantine Fast Grey NGLL—This direct dye gives neutral greys of good fastness to light on all types of cellulosic fibres. It is very level dyeing and is thus of interest not only alone but also as a shading component in combination with other Chlorantine Fast dyes. Dyeings become more olive-green when crease-resisted, but their fastness to light is unaffected and the fastness to washing improved. Fastness to wet treatments may be improved by aftertreating with Lyofix 8B conc. or EW, but the light fastness is slightly impaired. Chlorantine Fast Grey NGLL may also be aftercoppered. Fastness figures on cotton include—Light 6, washing 3-4, perspiration 4.

#### Compagnie Française des Matières Colorantes

The Neutrogene Printing Colours—This is a comprehensive monograph describing the printing properties and application of fourteen Neutrogene dyes, a range of azoic printing compositions which are applied by simple direct printing and are fixed by steaming for a short time under neutral conditions. Each member is illustrated by a small print in three depths, and the wide range of possible applications in machine and screen printing, alone and in conjunction with vat dyes, solubilised vat dyes, and Solanile Black, is indicated by the inclusion of eleven large patterns from bulk productions. Information given in the text includes details of the preparation and the storage of the printing pastes, printing recipes, and development together with notes on the types of fabric, possible styles, and the production of mixture shades. Screen printing and the detection and correction of faults are dealt with in separate sections.

are dealt with in separate sections.

In addition to the Neutrogene range, details are given of four supplementary dyes—Neuzogene Light Blue B, an azoic printing composition which can be applied under the same neutral-dyeing conditions as the Neutrogenes to give fast-to-light navies; and Sulphanol Fast Blacks CLA, CLB, and CLJ, which are sulphur dyes for textile printing recommended for outline blacks under azoic, solubilised vat, and vat dyes and under white discharges and illuminated discharges containing vat dyes.

#### Durand & Huguenin S.A.

Indigosol Grey 13F—This solubilised vat dye is particularly recommended for printing to give greys on cellulosic materials when maximum fastness to boiling

soap and soda is required. It can be reserved without the need to use special recipes, and very bright headings may be printed under fast-dyed grounds. It is readily dischargeable to a good white. Fastness figures on cotton include—Light 6-7, soda boiling (Test A on pattern printed by the nitrite process) 4-5, chlorine (Test A) 5.

Panduran Turquoise—This is a phthalocyanine derivative developed for use as a printing colour to give greenish blues of very good fastness to light, wet and dry rubbing, and chlorine. It is completely fixed by steaming, and no special aftertreatment is required. It may be mixed in all proportions with chrome dyes, and printed alongside vat dyes, solubilised vat dyes, and stabilised azoic combinations, with which it may also be mixed if development in neutral steam is used. Reserve styles under solubilised vats and aniline black are readily obtained. Fastness figures for a direct print developed by steaming for 20 min. include—Light 6, water 4–5, chlorine 5, soaping at 95°c. 3–4.

#### The Geigy Co. Ltd.

IRGALAN BROWN VIOLET DL— This is a premetallised dye of the neutral-dyeing type particularly recommended for use in mixtures for producing dark browns, greys, bordeaux, and navy blues. It is suitable for dyeing wool and polyamide fibres as loose stock, slubbing, yarn, and cloth where excellent fastness to light and very good fastness to wet treatments are required. Unlike most of the Irgalans, a distinct reddening of the hue occurs in artificial light. In addition to dyeings on wool fannel and nylon yarn and a white discharge on silk, twelve mixtures with other Irgalans are shown, each in four depths. Fastness figures on wool include— Light 6–7, heavy milling 4, perspiration 5.

IRGALAN DARK BROWN 5R—This neutral-dyeing premetallised dye gives full reddish browns on wool, polyamide fibres, and silk. The fastness properties of the dyeings obtained are slightly inferior to those given by the Irgalan L brands and it is therefore recommended for deep dyeings only. The hue becomes distinctly redder in artificial light, and it is suggested that this characteristic will prove useful when Irgalan Dark Brown 5R is combined with Irgalans Grey BL, Brown Violet DL, and Yellow 2RL for heavy fashion shades.

#### Imperial Chemical Industries Ltd.

Brentogen Brown B—This stabilised azoic dye combination is recommended for printing cotton, viscose rayon, and linen cloth to give browns of good fastness to light and wet treatments. It is applied from an alkaline printing paste and developed by acid steaming or treatment in an acid bath. If Brentogen Developer N is used, it is possible to develop in neutral steam and thus to print alongside vat dyes. It is also of value for printing alongside pigments, solubilised vat, basic, and mordant dyes, and also Alcian Blue 8G. Fastness figures on cotton include—Light 6-7, washing (repeated severe, 5 times at 100°c.) 3-4, hypochlorite ("dry chemicking") 4-5.

Brentogen Brown 2R.—This stabilised azoic combination is recommended for direct printing on all types of cellulosic fibres but particularly for the dress goods and overall trade. It is especially useful for use alongside Soledon range, from which deep browns are not readily obtained. Its printing characteristics and the properties of the prints obtained are similar to those of Brentogen Brown B. Fastness figures on cotton include—Light 6–7, washing (repeated severe, 5 times at 100°c.), 4–5, hypochlorite ("dry chemicking") 4–5.

DURINDONE PRINTING PINK 2B—This homogeneous vat dye gives bright bluish reds of very good fastness to light and washing on cellulosic materials. Fastness figures on cotton include—Light 6-7, washing (repeated severe, 5 times at 100°C.) 4, hypochlorite ("dry chemic") 5.

LISSAPOL GL PASTE—This is a neutral, non-ionic detergent containing dirt-suspending products introduced to provide a complete laundry detergent requiring no other additions in use except the correct amount of the prescribed alkali for the particular laundry classification to be washed. Washing formulæ for eleven different classifications are appended.

Resist Salt L.— This is a weak oxidising agent under alkaline conditions and is recommended as an addition to printing pastes and kier-boiling and mercerising liquors to prevent the adverse effect of the accumulation of reducing agents such as starches and decomposition products present in sizes and textile gums. Unlike chlorates, it is effective in small quantities and involves no risk of tendering of the material.

Shiblastains A and C for the Identification of Textile Fibres—This is a revised edition of the technical circular describing the use of two types of stain for fibre identification. The A type is designed to discriminate between a wide range of textile fibres, while Shirlastain C is recommended for distinguishing between cellulosic fibres and also particular forms of the same fibre, e.g. raw, secured, and bleached cotton. Shirlastain A is also of interest in the control of silk degumming, the removal of gelatin sizes, and the detection of hydrolysis in acetate rayon.

Soledon Brown R.—This solubilised vat dye is derived from Caledon Brown R and gives orange-browns of excellent fastness to light and wet treatments on cellulosic fibres. It is of particular interest alone and in mixtures for colouring highly twisted yarms and tightly woven fabrics by either a dyeing or a padding technique, the dyeing being developed by the normal nitrite-sulphuric acid method. It has high affinity for silk, to which it is preferably applied by the acid exhaust method followed by development by the thiceyanate-dichromate process. Soledon Brown R is very suitable for direct printing to give colours which have hitherto been obtainable only with difficulty from the parent vat dye. Dyeings or prints on cellulosic fibres do not undergo accelerated tendering on exposure to light. Fastness figures for a dyeing on cotton include — Light 6-7, washing (repeated severe, 5 times at 100°c.) 4-5, hypochlorite 5.

Soledon Red 2B— This homogeneous solubilised vat dye gives pinks and reds of good fastness to light and wet treatments on cotton, for which it has moderate affinity at 40°c. It may be applied, alone and in mixtures, to yarn in hank form, packages, knitted fabrics on the winch, and cloth by dyeing or padding on the jig. On viscose rayon good exhaustion may be obtained by using short liquor: material ratios and adding salt. Crease-resist finishing improves the fastness to light and has no effect on the hue, and good coverage is obtained on "barry" viscose rayon. It is also of interest for printing, particularly where good fastness to light and washing is required and where freedom from accelerated degradation of cellulose on exposure is essential. Fastness figures for a dyeing on cotton include—Light 5, washing (repeated severe, 5 times at 100°c.) 4, bypochlorite 5.

#### Sandoz Products Ltd.

AETISIL DIRECT ORANGE RFL—This disperse dye gives bright oranges on acetate rayon, similar in hue to those obtained with the Orange 2R, 3RP, and 4RP brands but having better fastness to light, gas-fume fading, sublimation, and wet treatments. On nylon, the colour obtained is redder and slightly duller. This dye is of interest for dyeing loose material, yarn, and cloth, alone and in mixtures, notably with the Blue GFL and Scarlet GFL brands where maximum fastness to light and gas fumes is desired. It may be applied to Terylene by normal dyeing techniques in the presence of a carrier, and is also of interest for dyeing at elevated temperatures in high-pressure dyeing machines. Fastness figures on acetate rayon include—Light 6-7, washing (40°c.) 4-5, gas fumes 5.

DIAZAMINE FAST SCARLET RWL—This direct cotton dye gives bright reds on cellulosic fibres when diazotised and developed on the fibre with  $\beta$ -naphthol. The dyeings obtained are brighter and redder than those given by the GL or GLA brands, faster to wet treatments than those from the other Diazamine Scarlets, and unaffected by crease-resist finishing. White discharges may be obtained under both neutral and alkaline conditions. Viscose rayon having irregular dyeing characteristics is dyed satisfactorily and solid dyeings may be obtained on cotton-viscose rayon mixtures. Fastness figures of a dyeing on cotton diazotised and developed with  $\beta$ -naphthol include—Light 4, washing (40°c., medium dyeing) 4–5, perspiration 5.

FAST-TO-WASHING COLOURS ON HOSIERY YARN—This card contains dyeings in two depths of a range of twenty acid dyes on wool yarn. When applied to either wool or chlorinated wool by the recommended dyeing procedure, the dyeings obtained will pass the S.D.C. Washing Test No. 2 (Wool), and the range is recommended for dyeing yarn, knitted fabric, and cloth where good fastness to washing is required. The majority of the dyes may be dyed under neutral or nearly neutral conditions. Pretreatment at 60°C. for 15 min. in a bath containing 3–5% ammonium sulphate or 3–5% ammonium dihydrogen phosphate is recommended to equalise the pH throughout the material. The dye is then added; the temperature raised to 80°C. during 20–30 min., held at 80°C. for 15 min., and raised to boiling point in 20–30 min.; and dyeing continued at the boil for 15 min. only.

LUMICREASE FAWNS AND GREYS—This card contains dyeings in four depths on viscose rayon staple cloth of Lumicrease Fawns RLF and GLF and Greys GLF and BLF. As the generic name suggests, these dyes are particularly suitable for dyeings which are subsequently to be given a crease-resistant finish, the fastness to light being maproved by half a point. Improved fastness to washing may be obtained by aftertreating with Sandofix WE, but the fastness to light is slightly reduced. Fastness figures include—Lumicrease Fawn RLF and GLF—Light 6 (crease-resisted dyeing), washing 3, perspiration 3-4. Lumicrease Grey GLF and BLF—Light (crease-resisted dyeing) 6, washing 3, perspiration 3-4.

Omega Chrome PL Colours—This is a range of eight chrome dyes compatible with each other and designed for use alone and in mixtures for deep dyeings on wool yarn which will withstand S.D.C. Washing Fastness Test No. 2 (Wool). For shading and brightening purposes the Xylene Fast P dyes are recommended. The following dyeing procedure is recommended for yarn—The yarn is entered into a liquor containing 10% Glauber's salt and sufficient acetic acid (approx. 2·5%) to give a pH of 5·0 ± 0·1, and treated at 72–73°c. for 15 min., after which the pH of the liquor is determined and adjustment made if necessary. The yarn is then lifted, and the dissolved dye added.

Dyeing is commenced at 70°c. for 15-20 min., the solution raised to boiling point in 20-25 min., and dyeing continued at boiling point for 30 min. The bath is cooled to 80°c., the dichromate added, chroming continued at 80°c, for 10 min., the temperature raised to boiling point in 10 min., and chroming continued at the boil for 20 min. It is emphasised that on no account should further acid be added after the initial pH has been adjusted to the correct value.

SOLAR YELLOW 3LG: CUPROFIX YELLOW 3LG: RESOFIX YELLOW 3LG—The versatility of this direct cotton dye is reflected by the number of names by which it is known. Thus it is of interest on cellulosic materials when dyed direct and also when aftertreated on the fibre with Sandofix WE, Cuprofix S new, and Resofix CU, the wet fastness of the dyeing increasing in that order.

hue of the direct dyeing becomes slightly redder and the light fastness is slightly reduced on crease-resist finishing. Fastness figures on cotton include—Direct dyeing: Light 6-7, washing (Test No. 2, 40 c.) 3-4. Dyeing aftertreated with Resoftx CU: Light 6-7, washing (Test No. 2, 40 c.) 5.

#### Town End Chemical Works Ltd.

AZONINE AND DURANTINE FAST COLOURS ON COTTON YARN-This card contains dyeings in three depths on cotton yarn of 56 direct cotton dyes, 38 of which are included in the Azonine range and the remainder in the Durantine Fast range. Comprehensive information on fastness properties is appended alongside each set of patterns, together with recommended uses. The dyeing characteristics of each dye have been determined according to the recommendations of the Society and each has been classified accordingly.

## Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

#### I-PLANT: MACHINERY: BUILDINGS

Drive Problems of Jiggers and Batching Devices.
G. Kadegge. Textil-Praxis, 9, 380-384 (April 1954).
Uniform treatment of fabric depends, amongst other factors, on constant cloth speed through the liquor. Constant tension on the batch also makes for even penetration and the avoidance of structural damage to delicate fabrics. Examples of constant speed-tension jiggers and their drives are described and treated mathematically, concluding with a machine driven by two D.C. variablevoltage motors, each of which may act as a drive or a generator. Constant cloth speed is obtained with the help of a tacho-dynamo. The principle is applicable to other batching machines. S. R. C.

Bond Machine for bringing Fabrics into Quick Contact with Dye and other Liquors. B. C. Bond. Amer. Dyestuff Rep., 43, P232-P235 (12 April 1954).

An experimental dyeing machine is described in which cloth passes in open width between, but without touching, parallel perforated metal plates, through which liquor flows, by way of systematically placed holes, perpendicularly towards both faces of the cloth and then laterally back to a Liquor speed is very high, e.g. 800 gal. (U.S.A.)/ min., thus, in effect, bombarding the cloth, which travels at 20-120 yd./min., and giving excellent levelness and penetration. The volume of liquor is small, e.g. 50 gal. penetration. The volume of liquor is small, e.g. 50 gar. (U.S.A.) for 60 in. width, and is maintained by gravity feed with automatic float control. Only two operators are required for a commercial range. Three units are satisfactory for applying soluble vats, direct, or disperse dyes, where no aftertreatment is required. A detailed procedure for vat dye application is given.

McKiernan-Terry Multipurpose Calender. J. W. Wilkinson. Amer. Dyestuff Rep., 43, 247-248 (12 April

A new multipurpose calender is briefly described.

PATENTS

Thread Processing Reel. Courtaulds. BP 709,873 A reel in which the thread is treated by both acid and C. O. C. alkaline hot solutions.

Cloth Insert for Rayon Cake before Wet Processing. American Enka Corpn. An expansible member formed of cloth and flexible stainless steel spring wires readily inserted into freshly spun rayon cakes as protection during subsequent wet processing and handling. C. O. C.

Shrinkable Paper Wrappers for Rayon Cakes or similar Packages. American Viscose Corpn. BP 709,605

The wrapper consists of a liquid-permeable paper or paper-like material coated in part with a moisture-proof or moisture-repelling elastomer. This coating which occurs

only at the outer side wall of the package, shrinks with the base sheet during drying to fit closely against the side wall of the package and retards evaporation of moisture from the outer windings during drying. C. O. C.

Tenter. Mather & Platt. BP 709.248 Simple and effective automatic means to vary the effective width of the hot air nozzle slots as fabrics of different widths are fed into the machine so that all the air discharged through the nozzle makes direct contact with the C. O. C.

Ensuring Constant Tension while Winding a Web. Timsons. BP 710,263

Garment Pressing Machines. Isaac Braithwaite & Son,

Paper Coating Machine. St. Regis Paper Co. USP 2,645,199

Paper Coating Machine. Combined Locks Paper Co. USP 2,645,201

#### II— WATER AND EFFLUENTS

Iron Treatment of Waste Liquors containing Dyes II. M. Kehren and J. Thewis. Melliand Textilber., 35, 183-185 (Feb.), 284-285 (March 1954).

Some experiments on the coagulation of Benzopurpurine 10B soln, and the electrophoresis of sols of Indigocarmine, Benzopurpurine 10B, and iron oxide are reported. H. E. N.

PATENTS

Extracting Wool Grease from Wool Washes. 709,567

Wool-wash liquor is passed into a container and treated at not > 40°c, with bubbled air under conditions which produce a grease-rich froth. This is passed to an adjoining compartment where, on gentle agitation at 80-90 c., it is converted to a liquid which may be economically treated for grease recovery by conventional methods. J. W. B.

#### III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Thermodynamic Quantities associated with the Interaction between Ethylenediaminetetraacetate and Alkaline-earth Ions. F. F. Carini and J. Amer. Chem. Soc., 76, 2153-2157 E. Martell. (20 April 1954).

Thermodynamic equilibrium constants for reactions between  $M^{z_+}(aq_+)$  and  $V^{z_-}(aq_+)$  ( $M^{z_+}=$  alkaline earth ion;  $V^{z_-}=$  tetranegative anion of ethylenediaminetetra-acetic acid) have been calculated from e.m.f. data obtained from cells of type Pt-H<sub>2</sub>,  $K^+(m_1)$ ,  $M^{2+}(m_2)$ ,  $Cl^-(m_2)$ ,  $H_nV^{n-4}(m_4)$ , AgCl-Ag at various temperatures and ionic strengths. For each temperature the data were extrapolated to infinite dilution with the aid of the Debye-Hückel activity

coefficient relationship, and the thermodynamic chelate formation constant evaluated. The thermodynamic quantities  $AF^{\circ}$ ,  $AH^{\circ}$  and  $AS^{\circ}$  are calculated and discussed in the light of present theories of metal chelate formation in aqueous solutions. The most significant result of the investigation is that the high stability of alkaline earth-ethylenediaminetetra-acetate chelates primarily to the large entropy increase associated with complex formation. C. O. C.

R. K. Her. Stearatochromic Chloride. R. I Chem., 46, 766-769 (April 1954). Ind. Eng.

Stearatochromic chloride (Quilon, DuP) is prepared by heating basic chromic chloride with stearic acid in methanol, and is thought to have the structure-

It is a water-soluble cation-active surface-active agent which is strongly adsorbed from aqueous solution by negatively charged surfaces, e.g. cellulose, proteins, glass-The adsorbed film is hydrophobic, and when dried becomes insoluble in water, probably owing to hydrolysis and polymerisation of the resulting basic chromic cations. Th product is employed as a water-repellent for paper, felt, and glass fibre, and as an antisticking agent. W. K. R.

#### PATENTS

## Washing and Cleaning Agents containing Alkali Salts of Carboxyalkyl Cellulose. N. V. Handelsvereeniging "Holland". BP 710,777

A cleaning washing, or soaking composition comprises one or more alkali salts of carboxyalkyl cellulose having a degree of substitution of 0.49-0.60 and a fibre content of fegree of such a constitution of 0.49-7-700 and a fibre content of 6-16%, and of such a constitution that a 1% by wt. solution of the salt in water, buffered by 2.5% by wt. of Na<sub>5</sub>CO<sub>3</sub> and 2.5% by wt. of NaHCO<sub>3</sub>, has a viscosity of 20-30 cP when measured with a standard Höppler viscometer at 20°C<sub>3</sub>, together with one or more other detergents. A typical composition contains 100 mg. of pure dry Na carboxymethylcellulose/l. of washing liquor and a quantity of fatty acid soap corresponding to 1 g./l. of fatty J. W. B.

## Detergent and/or Bactericidal Compositions. ICL BP 710,105

Compounds of formula-

(A and B = same or different, aromatic nuclei which may be substituted by one or more OH, Hal, NO<sub>3</sub>, Alk or alkoxy radicals; X = bydrocarbon or dialkoxybenzene of < 16C) have excellent bactericidal properties. They are mixed with detergents in the proportion of 10 parts by weight of the bis-biguanide to 5-2000 parts of detergent.

Fibre Lubricants. ICI. Aqueous solutions of the condensates of 1 mol. of an alkylphenol, preferably Alk of 6-20C, with 5-20 mol. of

ethylene oxide are used as fibre lubricants, e.g. in woollen spinning. Addition to the solution of \$12% by wt. of sodium benzoate and \$15% of a polyhydric alcohol, preferably glycerol, permanently inhibits it, causing rusting.

#### Quaternary Ammonium Naphthenate— A. M. Erskine. e-Antiseptic. USP 2,645,593 Compounds of formula-

(R = Alk of 8-18C) are self-emulsifying highly potent agents for inhibiting the growth of micro-organisms C. O. C.

#### Surface-active Phenois Condensates from Naphthols. Basf. 709,859

A phenol or naphthol which is not substituted by alkyl radicals containing > 3C is reacted with > 1 mol. propylene, or propyl or iso-propyl alcohol, per mol. of phenol or naphthol, followed by reaction with ethylene oxide or polyglycols to introduce water-solubilising groups.

## Production of Undecomposed Cellulose Oxidation Products, including their Alkali and Alkaline Earth Metal Salts, and their use as Detergents, Cation Agents, and Filter Aids, etc. Nederlandse Organisatio yoor Toegepast-Natuurwetenschappelijk Onderzoek ter Behoeve van Nijverheid, Handel en BP 709,941

Cellulose which contains lignin and/or resin is subjected to controlled oxidation, e.g. with HNO<sub>3</sub> and NaNO<sub>3</sub>, until the product contains 0.5–5.5, preferably 1.0–5.5, m. eq. of base-binding groups per g. of dry oxidation product, and, if desired, is converted to alkali or alkaline-earth metal salts. In one example oxidised deciduous wood shavings are used as an exchange water softener. In another, a are used as an exchange water softener. In another, a washing and cleaning agent with the consistency of soft soap, and particularly useful for applying as a paste to dirty spots on laundry, comprises Na salts of sulphonic acids of high mol. wt. hydrocarbons (30%), Na salts of high mol. wt. fatty acids (5%), NaCl (5%), Na<sub>2</sub>CO<sub>3</sub> (3%), Na salt of carboxymethyl cellulose (2%), oxidised straw (2%), and water to 100%. and water to 100%

## Organic Silicon Compounds useful as Auxiliaries.

Joseph Crosfield & Sons.

Joseph Crosfield & Sons.

RP 709,634

Compounds of formula (NR¹R³R³R³h¹)<sub>2</sub>O·x8iO<sub>3</sub>·yH<sub>2</sub>O

(R¹, R³, R³ and R⁴ = same or different H, Alk, hydroxy-alkyl, amino alkyl, amidino, cycloalkyl, alkaryl or aryl, but R<sub>4</sub> cannot be H or Alk; or R<sub>3</sub> and R<sub>4</sub> = -CH<sub>3</sub>- groups forming part of a heterocyclic ring structure which includes the N atom), have many industrial applications including wetting and emulsifying. A method of preparation comprises dissolving a silica which is soluble in aqueous alkalis in a solution containing at least 0.1% by wt. of an organic nitrogen base having a dissociation constant of at least 10-5, and recovering the resultant organic nitrogen J. W. B. base silicate.

### Sizes. Société "Rhodiacéta". A size comprises an aqueous solution of gelatin containing 0.5–30% by wt. of at least one salt of H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, or a lower saturated aliphatic carboxylic acid, together with a saturated aliphatic hydroxyamine of < 7C. In one example 5 kg. gelatin swell in 10 kg. water for 12 hr., after which the mixture is heated to 55°c. to dissolve the gelatin, and 4.3 kg. of ethanolamine nitrate and 85 kg. of water are added, and stirred. At 55°c. it is an excellent size for 100

## Melamine Resins-Textile Finishes and Adhesives.

J. W. B.

den. cellulose acetate.

Melamine with 5-35% urea is condensed with formaldehyde in aqueous methanol or ethanol until the product gives a clear solution with > an equal vol. of water. The products are useful as adhesives and for impregnating

Fungicides and Bactericides. Anorgana. BP 708,963
The esters formed by aliphatic carboxylic acids of < 9C with polyhydrio, preferably aliphatic, alcohols or their halogenohydrins are suitable for incorporating into paints, distempers etc., or for impregnating paper or regenerated cellulose film to prevent the growth of microorganisms.

#### Water-soluble Asymmetric Ureas and Thioureas Mothproofing Agents. Variapat.

The asymmetric ureas prepared from halogen-containing aminosulphonic acids of the benzene series having a sulphonic acid group in o-position to the amino group with halogen containing reactive carbonic acid or thiocarbonic acid derivatives in which the final condensate contains at least one trifluoromethyl group and a sulphonic acid group in o-position to the acylated amino group, e.g.-

are mothproofing agents and give protection to all types of fibres against textile pests. C. Ö. C.

Finishing Agent for Laundered Garments. J. G. Wiegerink. USP 2,645,584

A composition consisting essentially of a polyoxyethylene glycerol triricinoleate and/or trihydroxystearate
and a water-soluble to colloidally dispersible polymeric
colloid, e.g. carboxymethylcellulose, can very well replace
starch as a finishing agent for laundered garments. It
enables the garments to be pressed or ironed at a higher
temperature than usual.

C. O. C.

Tanning Agents. O. C. Jekel. BP 709,581
Pure vegetable tanning agents are treated with boric acid in a mutual organic solvent and the solvent evaporated to yield the resultant solid compound. The products are stronger acids than either of the two components, have much higher tanning power than the original tannin and form particularly stable solutions.

C. O. C.

Reserving Nylon against Acid and Mordant Dyes when mixed with Wool (VIII p. 374).

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Continuous Production of Nitrotoluenes. F. Meissner, G. Wannschaff, and D. F. Othmer. Ind. Eng. Chem., 46, 718-724 (April 1954).

A description is given of a process used in Germany for the continuous production of mono- and tri-nitrotoluene. A combination of parallel flow and counter flow of nitrating acid and toluene is employed. Measured streams of toluene and mixed nitrating acid are separately fed into the main nitrator where conditions are controlled so that nitration is substantially complete, and the product passes into second and third nitrators, in parallel flow, where the reaction is completed. The mononitrotoluene containing 1% toluene flows into a continuous decanter where the spent acid is separated, the nitrotoluene passing on to continuous washing units. The spent acid flows into a tank along with fresh toluene which removes any residual nitric acid, and after separating from the final spent acid, the mixture of toluene and nitrotoluene is passed into the main nitrator and the acid is concentrated. All equipment is of stainless steel and no centrifugal pumps are required. Trinitrotoluene is prepared similarly in three nitrations. The advantages of continuous over batch production are discussed. W. K. R.

Nitration of 2-Methylnaphthalene. J. A. Brink and R. N. Shreve. Ind. Eng. Chem., 46, 694-702 (April 1954).

Mononitration of m-Xylene. K. A. Kobe and H. M. Brennecke. Ind. Eng. Chem., 46, 728-732 (April 1954).

Brennecke. Ind. Eng. Chem., 46, 728-732 (April 1904).

A maximum yield of 98% mononitro-m-xylene is obtained by nitrating under the following optimum conditions—1-08 moles of sulphuric acid per mole of m-xylene; sulphuric acid concentration 81%; 10% excess nitric acid; at 30°c. for 60 min. The product consists of 4-nitro-m-xylene and 2-nitro-m-xylene in the satio 85:15. Above 50°c. there is a complete absence of dinitro-m-xylene, but there is tar formation. Of the three xylenes, m-xylene is the most easily nitrated.

W. K. R.

Syntheses in the Naphthalene Series — Methoxylated Tetralones and Naphthols. O. Brunner and P. Hanke. Monatsh. Chem., 85, 88-91 (Feb. 1954).

The title compounds are prepared by condensation of phenol ethers with succinic anhydride, reduction of the keto acids formed by Lock's method (Osterr. Chem. Ztg., 51, 77 (1950)), and cyclisation of the resulting phenyl butyric acid deriv. to the corresponding tetralone, whence dehydrogenation affords the naphthol. A second procedure is via the condensation of quinol dimethyl ether with ethylsuccinic anhydride. The preparations of 5:7:8-trimethoxy-1-oxo-1:2:3:4-tetrahydronaphthalene, 5:7:8-trimethoxy-1-naphthol, 5:6:8-trimethoxy-1:2:3:4-tetrahydronaphthalene, 2-ethyl-5:8-dimethoxy-1-oxo-1:2:3:4-tetrahydronaphthalene, and 2-ethyl-5:8-dimethoxy-1-naphthol are described. H. H. H.

Kinetics and Mechanism of Diazotisation—VII.
H. Schmid. Monatsh. Chem., 85, 424-440 (April 1954).
The general reaction mechanism is established for

The general reaction mechanism is established for aromatic, aliphatic, and inorganic diazotisation in any given acid, and special cases are discussed. The velocity of the reaction—

$$H_2NO_3^+ + NO_2^- \rightarrow N_2O_3 + H_2O$$

at 0°c, is found to be  $3\times10^{6}$  times greater than that of—  $H_{4}NO_{3}^{+}+NO_{3}^{-}\rightarrow N_{3}O_{4}+H_{4}O$ 

The partial reactions with  $H_2NO_3^+$  are introduced into the  $HNO_3-HNO_3-NO$  reaction as follows—

$$HNO_{2} \rightleftharpoons NO_{1}^{-} + H^{+}$$
  
 $HNO_{3} + H^{+} \rightleftharpoons H_{4}NO_{2}^{+}$   
 $H_{2}NO_{3}^{+} + NO_{3} \rightleftharpoons N_{4}O_{3} + H_{2}O$   
 $N_{2}O_{2} \rightleftharpoons NO_{2} + NO$   
 $2NO_{2} \rightleftharpoons N_{2}O_{4}$   
 $N_{4}O_{4} + H_{4}O \rightleftharpoons NO_{3}^{-} + H_{2}NO_{4}^{+}$ 

The velocity coefficient of the hydrolysis-

NOBr +  $H_2O \rightarrow HNO_2 + H^+ + Br^-$ 

is evaluated from diazotisation data as  $8\times10^6$  at  $0^\circ\mathrm{C}$ . The velocity coefficients of the nitrosation of aromatic, aliphatic, and inorganic amines (in non-ionisable form) by  $N_3O_3$ , NOCl, NOBr, and NOHSO $_4$  are calculated and compared. 50 References. H. H. H. H.

Aromatic Diazo Compounds. XIII — Oxidation of Azo Dyes with Air. J. Poskočil and Z. J. Allan. Chem. Listy, 47, 1801–1810 (1953): Chem. Abs., 48, 4221 (10 April 1954).

Azo dyes prepared by coupling 1-diazo-2-naphthol-4-sulphonic acid with various components were oxidised with air in alkaline solns, containing catalytic amounts of Cu or Mn salts. When the coupling component was m-phenylene diamine, triazoles were formed; dyes derived from aceto-acetanilide gave 1:2:3:5-triazolones; when the azo dye had no NH<sub>2</sub> ortho to the azo group the products of oxidation included a diazo compound and a quinone. Thus the monoazo compound 1-amino-2-naphthol-4-sulphonic acid -> resorcinol gave 4-diazoresorcinol and 2-hydroxy-1:4-naphthoquinone. The dye p-aminobenzoic acid -> aceto-acetanilide was unchanged by the air oxidation treatment. E. S.

Azo Dyes from Anilides of Diphenic Acid. B. M. Krasovitskii and E. 8. Khotinskii. Ukrain. Khim. Zhur., 18, 488–503 (1952): Chem. Abs., 48, 4839 (25 April 1954).

The acid chloride of diphenic acid was condensed with m- and p-nitroaniline, followed by reduction, to yield the diarylides—

The monoarylides were made similarly from diphenic anhydride. These compounds were di- and tetr-azotised and coupled with various naphthol- and aminonaphthol-sulphonic acids to give acid dyes, and with arylides of 3:2-hydroxy-naphthoic acid, 3-hydroxy-phenanthrene, etc., to give dyes of the azoic type. The dyes so produced were in general similar to those obtained in a similar way from m- and p-aminoanilides of benzoic acid.

E. S.

Azo Dyes. IV—Dyes derived from 5-Amino-2-naphthol-7-sulphonic Acid. K. Murata and K. Harada, Bull. Fac. Engr., Hiroshima Univ., 2, 233-6 (1953): Chem. Abs., 48, 5501 (10 May 1954).

Synthesis from Na 5-amino-2-naphthol-7-sulphonate is described of the following 12 dyes (hues on wool)—8-amino-2-(6-hydroxy-3-sulpho-1-naphthylazo)-1-naphthol-3; 6-disulphonic acid, dark purple; 5-(6-amino-1-hydroxy-3-sulpho-2-naphthylazo)-2-naphthol-7-sulphonic acid, dark red; 5-(7-amino-1-hydroxy-3-sulpho-2-naphthylazo)-2-naphthol-7-sulphonic acid, redish purple; 5-(2-amino-8-hydroxy-6-sulpho-1-naphthylazo)-2-naphthol-7-sulphonic acid, dark red; 5-(2-hydroxy-7-sulpho-1-naphthylazo)-2-naphthol-7-sulphonic acid, brown; 8-(5-amino-2-hydroxy-7-sulpho-1-naphthylazo)-1-naphthol-3-sulphonic acid, purple; 6-(5-amino-2-hydroxy-7-sulpho-1-naphthylazo)-1-naphthol-3-6-disulphonic acid, purple; 6-(5-amino-2-hydroxy-7-sulpho-1-naphthylazo)-1-naphthylazo)-1-naphthol-3-6-disulphonic acid, purple; 6-(5-amino-2-hydroxy-7-sulpho-1-naphthylazo)-1-naphty

sulpho-1-naphthylazo)-1-naphthol-3-sulphonic acid, deep red from acid bath; 7-(5-amino-2-hydroxy-7-sulpho-1naphthylazo)-1-naphthol-3-sulphonic acid, dark reddish purple; 5-amino-1-(4-sulpho-1-naphthylazo)-2-naphthol-7sulphonic acid, yellow; 5-amino-1-(4-nitrophenylazo)-2naphthol-7-sulphonic acid, orange. C. O. C.

Absorption Spectra and Molecular Structure— Analogous Behaviour of Azo and Imino Groups. A. A. Kharkharov. Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 117-123 (Jan.-Feb. 1954).

Evidence cited from the literature indicates that -O-, -S-, -NH-, and the like can be interpolated into cyclic and straight-chain systems of alternating double bonds without breaking the conjugation. This hypothesis provides an explanation of the deep colour of hydrazones of acridine-9-aldehyde hydrochloride (I), which cannot be explained on the assumption that they have the azo structure II, since the absorption spectra of the phenylhydrazone and the ethyl(phenyl)hydrazone (which cannot have the azo structure) are now shown to be practically identical. Further evidence is provided by the absorption spectra (in alcoholic soln.) of other compounds of formula I (X = OCH<sub>3</sub>, NO<sub>2</sub>, C<sub>4</sub>H<sub>5</sub>, N·N-, CH<sub>5</sub>O·C<sub>4</sub>H<sub>5</sub>, N·N-, C<sub>4</sub>H<sub>5</sub>, N·N-, and CH<sub>9</sub>O·C<sub>4</sub>H<sub>5</sub>, N·H-); the spectra of the first four of these are very similar in character to that of I (X = H) and differ greatly from the spectra of the last two, which have absorption bands at the extreme red end of the spectrum. The dye III (absorption max. 570 m\(mu\), cf. Safranine T, 535 m\(mu\), previously prepared by the author, is found to be substantive to cellulose fibres—an indication, it is considered, of the presence of a long, unbroken conjugated chain on the molecule. (Abstractor's Note—The author's formula is quoted below, although the fact that the dye is compared directly with Safranine T suggests that the phenyl group should be attached to the other N of the phenylaron.

Fluorescent Whitening Agents. I—Derivatives of Dichlorodiaminostilbenedisulphonic Acid. D. W. Hein and E. S. Pierce. J. Amer. Chem. Soc., 76, 2725-2731 (5 May 1954). Several fluorescent brightening agents have been propared from 5:5'-dichloro- and 6:6'-dichloro-4:4'-diaminostilbene 2.2' disuphynomic acid.

Several fluorescent brightening agonts have been prepared from 5:5'-dichloro-and 6:6'-dichloro-4:4'-diamino-stilbene-2:2'-disulphonic acid, mainly by acylation with substituted benzoyl chlorides. Their fluorescent properties and ultraviolet absorbency characteristics are compared with those of the corresponding acyl derivatives of 4:4'-diaminostilbene-2:2'-disulphonic acid and the influence of constitution in these properties discussed. C. O. C.

Molecular Conductivity of Dyes in Solution—I. K. Chaudhary. Pakistan J. Sci. Research, 4, 114-117 (1952) (in Pakistan J. Sci., 4): Chem. Abs., 48, 4938 (10 May 1954).

Molecular conductivities of several dyes in aqueous and alcoholic solutions were determined by a bright Pt electrode method (Indian J. Phys., 22, 1 (1948)). The conductivities

are not linear with the square root of concentration. The curves are similar to those obtained by Howell and Robinson for aqueous Na dodecyl and Na hexadecyl sulphates (Chem. Abs., 30, 6286 (1936)) and the departure from linearity is explained on the basis of formation of ionic micelles with increased concentration. The actual concentration at which the micelles form coincides with the concentration above which rapid quenching of fluorescence begins; this supports the view that the quenching results from ionic micelle formation.

C. O. C.

Hydroxyfuchsone Dyes. XIX—Effect of Nitro and Halogeno Substituents on the Indicator Properties of 4'-Hydroxy-3:3'-dimethoxyfuchsone. XX—Effect of Nitro and Halogeno Substituents on the Colour of 4'-Hydroxy-3:3'-dimethoxyfuchsone. I. S. Ioffe and B. G. Belen'kii. J. Gen. Chem. U.S.S.R., 24, 343-362, 353-361 (Feb. 1954).

A long discussion of the effects of the introduction of nitro and halogeno substituents into the previously unsubstituted phenyl group of 4'-hydroxy-3:3'-dimethoxy-fuchsone on the variation with pH of the position of the equilibrium between the numerous possible forms of the dye and its carbinol and on the absorption spectra of some of these forms.

A. E. S.

Investigations in the Naphtho[1,2]furazan Series. II—Nitration and Chlorination of Naphtho[1,2]furazan. III—Naphtho[1,2]furazan Bisulphite Compound and 7-Nitronaphtho[1,2]furazan. S. V. Bogdanov and S. F. Petrov. J. Gen. Chem. U.S.S.R., 24, 385-390 (Feb.); 532-535 (March 1954).

24, 383–390 (Feb.); 532–330 (March 1995).

Nitration of naphtho[1,2]furazan under conditions stated by Green and Rowe (J.C.S., 113, 67 (1918)) to lead to the formation of a single product (which they considered to be either 6- or 9-nitronaphtho[1,2]furazan (Ring Index numbering)) is found to give two mononitro compounds (6- and 8-), and also some dinitro cpd. The mononitro compounds are reduced to the amines, which are converted via the diazo cpd. into the corresponding chloro, hydroxy, and (in the case of the 6-amino cpd.) sulpho deriv. Direct chlorination of naphtho[1,2]furazan in presence of iron yields the 4-chloro deriv.; also, this and the 5-chloro deriv. are prepared by treating solutions of the corresponding sulphonic acids in hydrochloric acid with potassium hypochlorite. Various derivatives of the above compounds are described.

Naphtho[1,2]furazan bisulphite cpd. (4:5-dihydronaphtho[1,2]furazan-5-sulphonic acid) is prepared by the action of Na<sub>2</sub>CO<sub>3</sub> on the bisulphite cpd. of 1:2-naphthaquinone dioxime, and is then nitrated. Alkaline treatment of the 7-nitro cpd. formed yields 7-nitronaphtho[1,2]furazan, with elimination of sulphite, and this cpd. is converted into the corresponding amine, and hence, via the diazo cpd., into the corresponding chloro, hydroxy, and sulpho derivatives.

A. E. S.

"Pyridine" Dyes derived from Diphenyl. N. E. Grigor'eva and I. K. Gintse. J. Gen. Chem. U.S.S.R.,

24, 169-174 (Jan. 1954).

Reaction of 1-(2:4-dinitrophenyl)pyridinium chloride with 4-aminodiphenyl or one of its derivatives (benzidine, 4-amino-4-nitrodiphenyl, or 1-(4-aminodiphenyl-4-yl)-pyridinium chloride) yields dyes of formula—

The respective absorption max. (in acidic alcoholic soln.) are at 522, 526, 530 and 510 m $\mu$ . On being heated, III and IV decompose in the manner previously observed in dyes of this type, giving a pyridinium salt (e.g. 1-(4'-nitrodiphenyl-4-yl)pyridinium chloride from III) and the original amine (4-amino-4'-nitrodiphenyl from III), but I

and II isomerise; e.g. the product from I is the hydroehloride of-

A. E. S.

Synthesis and Properties of Azamethins derived from Weakly Basic Aromatic Amines. B. A. Poraï-Koshits and A. L. Remizov. J. Gen. Chem. U.S.S.R., 24, 372-375 (Feb. 1954).

Formation of azamethin compounds from amines and aldehydes is a reversible process, and in the case of weak bases, such as p-nitroaniline, the equilibrium is greatly in favour of hydrolysis of the azamethin, even in neutral soln. Such azamethins may be conveniently prepared in boiling toluene or xylene, water being removed as formed by azeotropic distillation. A. E. S.

Cyanine Dyes. VI—Syntheses of Aminovinyl Compounds by Addition of Amines. H. Zenno. J. Pharm. Soc., Japan., 73, 589-592 (1953): Chem. Abs., 48, 5857 (25 May 1954).

Production of some 35 compounds, e.g. the green 2-C<sub>6</sub>H<sub>5</sub>NHCH:CH·C<sub>5</sub>H<sub>5</sub>N·C<sub>2</sub>H<sub>5</sub>I, is described. C. O. C.

Preparation and Properties of Indo-oxine. W. Prodinger and O. Svoboda. Monatsh. Chem., 85, 23-28 (Feb. 1954).

Berg and Becker's preparative method for indo-oxine (I) by the action of NH<sub>2</sub>OH on 8-hydroxyquinoline (II) (cf. Ber., 73, 172 (1940)), which was used as a qual. test for NH<sub>2</sub>OH, is shown to afford a mixture of I with II. An improved product is obtained by the condensation of 5-amino-8-hydroxyquinoline with II. The absorption spectra of I in ethanol, benzene, acetone and dioxan, are described, together with the preparation of the hitherto unknown hydrochloride.

H. H. H.

Colours of Quinones and their Reduced Forms. E. Cerutti and J. Martinet. Teintex, 19, 325-350 (May 1954).

Numerous examples of quinones are considered, leading to the conclusion that the colours of such compounds, compared with the corresponding hydrocarbons, are deeper as R increases, where

$$R = \frac{N_{\rm h} + N_{\rm q}}{N_{\rm h} - N_{\rm q}}$$

 $(N_{\rm A}={
m number}~{
m of}~{
m possible}~{
m Kekul\'e}~{
m for}~{
m corresponding}$ hydrocarbon;

- number of possible Kekulé forms for quinone (idem,  $N_q = \text{number of possible 15}; ibid., 19, 277 (April 1954) ).$ 

Vat Dyes from 2:3-Dichloro-1:4-naphthaquinone by Condensation with Naphthols and their Deriva-

tives. B. Suryanarayana. Bombay Technologiat, 3, 83-84 (1952-53): Chem. Abs., 48, 5501 (10 May 1954). 2:3-Dichloro-1:4-naphthaquinone readily condenses with 1:2-benzobrazanquinone-4-carboxanilide to yield a dye which because of the presence of the carboxanilide group has good affinity for cellulose. Dyes similarly made containing heterocyclic rings, halogen atoms, and carboxamide and methoxy groups also confer affinity for cellu-lose. The range of hues obtained varied from yellow to violet. Condensing 2:3-dichloro-1:4-naphthoquinone with

 $\rm CH_{3}COOC_{3}H_{5}$  or acetoacetarylides in presence of pyridine and sodium ethoxide yielded 2-pyridinium 3-substituted 1:4-naphthaquinone betaine vat dyes, some of which dye cellulose orange reds. Their properties suggest a meso-ionic character. C. O. C.

Formation of Melanin from 5:6-Dihydroxyindole. J. M. Bruce. Chem. and Ind., 310-311 (13 March 1954).

The first stage in the oxidation of 5:6-dihydroxyindole to melanin is regarded as the formation of I, which on further oxidation gives II or III. Oxidation of the hydroxyl groups on either of these, but most probably on III, gives an orthoquinonoid system, which is considered to be capable of oxidative self-condensation to a melanin-like polymer.

Structure of Melanin. J. Harley-Mason and Bu'Lock, Chem. and Ind., 377 (27 March 1954). J. Harley-Mason and J. B.

3:5:6-Trihydroxyindole has been prepared in soln. and on oxidation gives a black ppt., soluble in aqueous alkali and thought to be 5:6:5':6'-tetrahydroxyindigo and not melanin as suggested by Stoves (ibid., 158 (1954)), although Stoves states that melanins from 5:6-dihydroxy indole are soluble in alkaline hydroxides. 3:5:6-Tri-hydroxy-1-methylindole cannot be oxidised to the 5:6quinone, which is not, therefore, regarded as a possible intermediate in the formation of adrenaline black (Stoves, loc. cit.). The 3-position of 5:6-dihydroxyindole is thought to be an essential point for condensation in the formation of melanin, rather than a site for hydroxylation and subsequent cross-linking (Stoves, loc. cit.). A. J.

Crystalline Chlorophyll-Protein Complex from Chlamydomonas. H. S. A. Sherratt and W. C. Evans. Nature, 173, 540 (20 March 1954).

Cells of Chlamydomonas were suspended in a mixture of inorganic medium and a-picoline and centrifuged. Addition of dioxan and storage at 0°c, caused separation of green crystals of chlorophyll-protein complex. Chlorophyll was extracted from the complex by organic solvents and absorption spectra showed the presence of chloro-phylls a and b. Amino acids were detected by paper chromatography of a hydrolysate of the complex. W. R. M.

Natural Anthocyanins and the Colour of Flowers. K. Hayashi. Kaguku (Science), 21, 400-4 (1951): Chem. Abs., 48, 5297 (10 May 1954). Review, 36 references.

Partition Chromatography of Tannins and Pigments. XII.— A Flavanoid Pigment Astragalin (Kaempferol-3-glucoside) in Astragalius sinicus Flowers. T. Nakabayashi. J. Agr. Chem. Soc. Japan, 26, 539-541 (1952): Chem. Abs., 48, 5942 (25 May 1954).

The name astragalin has been given to a new glycoside obtained from the flowers of A. sinicus. Its constitution has been shown to be kaempferol-3-glucoside. C. O. C.

Infrared Spectra of Gossypol. R. T. O'Connor, P. Von der Haar, E. F. DuPre, L. E. Brown, and C. H. Pominski. J. Amer. Chem. Soc., 76, 3268-2373 (5 May 1953)

The infrared spectra of chloroform solutions of gossypol and its derivatives confirm that it is an aromatic and phenolic compound. They also confirm presence of a carbonyl ortho to a hydroxyl group and in some derivatives of gossypol absence of carbonyl groups and in others presence of carbonyl groups directly attached to an alicyclic grouping. Thus the spectra strongly support the chemical evidence that gossypol occurs in tautomeric forms. These observations are in agreement with the accepted structure of the gossypol molecule described by Adams et al. But no evidence was obtained from the infrared data regarding the dinaphthyl character of gossypol nor of the presence of an isopropyl group.

C. O. C.

Green Earth as Substitute for Highly Coloured, Light-fast, and Alkali-stable Paint Pigments. F. Rösler. Chem. Tech. (Berlin), 5, 592-594 (1953): Chem. Abs., 48, 5518 (10 May 1954).

The Green Earths mined near Preisen and Taubenheim absorb basic dyes to give bright, stable, fast-to-light pigments suitable for use in paint and cement. C. O. C.

#### Amino- (or Hydroxy-) nitro-aryl-Alkylolamines-Hair Dyes, Lever Bros, and Unilever Ltd.

Arylalkylolamines containing nitro and amino (or hydroxy) groups are yellow to red hair dyes of good solubility in water. An example is—

which is made by heating 2:4-dinitrochlorobenzene with monoethanolamine in isopropyl alcohol at  $70-80^{\circ}\mathrm{C}$ . for 4 hr. After cooling and filtering, the product is reduced with sodium polysulphide in 50% aq. isopropyl alcohol at  $70^{\circ}\mathrm{C}$ . R. K. F.

#### Metallisable Monoazo Dyes for Wool. Ciba.

2-Amino-4-nitrophenol-6-sulphonamides (BP 709,495), which may carry an N-alkyl or N-aryl group in the sulphonamide group, are diazotised and coupled with a coupling component free of SO<sub>2</sub>H and COOH groups and capable of coupling ortho to an OH group, to give monoazo dyes which may be metallised (especially with Cr) in substance, on the fibre, or in the dyebath. Thus 2-amino-4-nitrophenol-6-sulphonamide is diazotised and the sparingly soluble diazo compound filtered off and coupled with an alkaline solution of 3-methyl-1-phenyl-5-pyrazolone. The product dyes wool yellowish-orange by the metachrome process. E. S.

#### Metal-complex Azo Dyes for Wool etc. Ciba.

The oo'-dihydroxymonoazo compounds sulphonamide or alkylsulphone of o-aminophenol → 1-alkyl (of > 3C)·3-methyl·5-pyrazolone (BP 697,056; J.s.D.C., 69, 462 (1953)), containing no SO<sub>3</sub>H or COOH groups, are treated with Co or Cr compounds, using at least half but less than 1 atom of metal per mol. of monoazo compound, to yield soluble products which dye animal and polyamide fibres from neutral or weakly acid baths. Similar products are obtained by metallising a mixture of a monoazo compound of the above type and a similar one containing e.g. a nitro instead of an alkylsulphone or sulphamyl group. Thus, all parts being by weight, the monoazo compound (35·2) 2-aminophenol·5-sulphonamide → 1-n-butyl·3-methyl·5-pyrazolone is refluxed for 6 hr. with an aq. soln. (125) of sodium chromosalicylate containing 2·6% Cr. The product dyes wool orange from a neutral or acetic acid bath.

#### Azoic Dyes for Mixtures of Wool with Viscose etc. FH. BP 709.666

Diazotised 4-halogeno-2:5-dimethoxyanilines coupled, on mixtures of wool and staple fibres of regenerated cellulose, with arylides of 3:2-hydroxynaphthoic acid, 3:2-hydroxycarhazole carboxylic acid, or o-hydroxybenzcarbazole carboxylic acid give bluish clarets to violet—

browns and dark violets of good rubbing fastness. For example, a mixed yarn of wool and staple fibres of regenerated cellulose is impregnated with an alkaline solution of the 4-chloro-2-methylanilide of 3:2-hydroxynaphthoic acid and then treated with a soln. of diazotised 4-chloro-2:5-dimethoxyaniline, to give a dark claret.

E. S.

## 2:5-dimethoxyaniline, to give a data distribution by the Blue, Copperable, Disazo Direct Cotton Dyes. Gy. BP 710,023

Blue direct cotton dyes suitable for aftercoppering are made by coupling tetrazotised benzidine-3:3'-diglycollic acid with an N-p-acylaminophenyl-J acid. The acyl radical may be derived from a monobasic aliphatic or benzene-series carboxylic acid or from a monoester of carbonic acid. An example is N-p-carbomethoxyethoxy-aminophenyl-J acid, which gives the blue direct cotton dye—

## Metal(copper)-complex Disazo Direct Cotton Blues

Tetrazotised dianisidine is coupled with 1 mol. of 1-naphthol-4:8 (or preferably 3:8) disulphonic acid and 1 mol. of an N-p-acylamino-phenyl (or -diphenyl)-1 acid to give disazo compounds whose copper complexes are bright blue direct cotton dyes. The p-acyl group may be an aroyl group but is preferably derived from a fatty acid of < 9C or a carbonic half ester of an alcohol of < 9C. Thus the disazo compound produced by coupling tetrazotised dianisidine first with 1 mol. of 1-naphthol-3:8-disulphonic acid in presence of Na<sub>2</sub>CO<sub>3</sub> and then with an ammoniacal solp of

is heated with ammoniacal copper sulphate, to give a complex which dyes cellulose fibres vivid blue. E. S.

## Azo Dyes containing an isoCyanate Group. DuP. USP 2,643,250

Dimers of aromatic isocyanates containing a ring structure D, which may be

are applied to textile fibres, paper, or leather and rendered fast to washing by heating to 120–200°c. This treatment is believed to regenerate –NCO groups which become firmly attached to reactive sites in the substrata, e.g. OH groups in cellulose. The dimer may be an azo dye, or it may be an amine capable of diszotisation and development on the fibre. Thus p-nitrophenylisocyanate, prepared from p-nitroaniline and phosgene, is dissolved in nitrobenzene and a little dimethylphenylphosphine added. After 4 hr. at room temperature crystals of—

separate. Reduction with H<sub>2</sub> in presence of palladium gives the corresponding diamine, which is tetrazotised and coupled with 3-methyl-1-phenyl-5-pyrazolone to give the disazo compound. A cotton fabric is soaked in water, wrung out, soaked in acetone, wrung out again and soaked in pyridine, which removes water from the fibres and leaves them in a swollen condition. It is then refluxed in a solution in dimethylformamide of the disazo compound, which dyes it yellow, fast to soaping.

E. S.

## Metallisable Direct Cotton Disazo Dyes. Ciba. BP 710,734

Aminomonoazo compounds of the constitution carboxyanilide of o-aminophenol→N·p-aminobenzoyl-J acid are diazotised and coupled with 8-hydroxyquinoline or a derivative (e.g. 5-chloro- or 7-sulpho-8-hydroxyquinoline) to give disazo direct cotton dyes which may be metallised

(especially with Cu) in substance, on the fibre, or in the dyebath. The carboxyanilide grouping, which may carry substituents (e.g. a sulpho group), renders the aftercoppered dyeings faster to light than the similar dyes of BP 586,840 (J.s.D.C., 64, 197 (1948)). Thus 2-aminophenol-4-carboxyanilide is diazotised and coupled with N-p-aminobenzoyl-J acid in presence of Na2CO3, and the product is diazotised and coupled with 8-hydroxyquinoline to give-

which dyes cotton red by the single- or two-bath aftercoppering process.

Trisazo Direct Cotton Dyes for Diazotisation and Development on the Fibre. FBy. BP 710,808
Trisazo dyes of the type subst. aminoazobenzenesulphonic acid -> N.-p-aminobenzoyl-J acid -> 2-amino-5naphthol-1:7-disulphonic acid dye cellulose fibres red to bluish violet, which may be diazotised and developed on

the fibre with  $\beta$ -naphthol to give bluish reds to reddish blues, clearer in tone than the dyeings given by known similar dyes in which the final component is J acid. Thus, by using as the first component the aminomonoazo m-4-xylidine-5-sulphonic acid -> p-cresidine, compound the trisazo dye-

is formed. It gives very clear violets when diazotised and developed in cotton with  $\beta$ -naphthol.

Azo Dyes. B. Gaspar and P. D. Dreyfus.

USP 2,644,753

Dyes containing at least one radical of formula-

$$(-N : N \cdot Y \cdot NZ)_{x-1} - B \xrightarrow{(OH)_{b-x}} NZ \cdot R \xrightarrow{(N:N-)} (SO_3 \text{ cation})_m$$

 $(B(OH)_{b-2} = x$  valent residue of a b-basic organic acid  $B(OH)_b; x = > 1; b \leqslant x; Z = H$ , Alk or Ar; R = napht-balene nucleus; m = 1 or 2; Y = bivalent aromatic radical; the azo groups above are part of a total of n' azo groups in the complete dye molecule and are arranged in n dye units separated from each other by heteronuclear bonds each unit containing—

$$a=\frac{n'}{n'}$$
 azo groups  $(a=1$ –4;  $n'=a.n$  and  $>b; n=>1)$ , are useful in colour photography. C. O. C.

BP 707,705 Blue Sulphur Dyes. Gy. Blue sulphur dyes of relatively high fastness to chlorine are made by sulphurising indophenols of formula-

(R = Alk of > 4C) or the corresponding leuco compounds. Thus the leuco-indophenol prepared from o-chloro-N-methyldiphenylamine is heated at 110°c, for 48 hr. with a mixture of S, Na2S and ethylene glycol monomethyl ether; the solvent steam-distilled off, and the dye precipitated by Ř. K. F.

Sulphur-containing Vat Dyes from Anthrimides

707,602 Anthrimides containing one anthraquinonyl and one ring system containing 6 condensed benzene nuclei, e.g. anthranthronyl, acedianthronyl etc., are treated with sulphur halides or halogen compounds of sulphur acids in fused AlCl<sub>3</sub>-alkali metal mixtures to obtain olive to red brown vat dyes. Thus the condensation product of 2aminoanthraquinone (I mol.) and dichloroacedianthrone (1 mol.) is added to a melt of AlCl, NaCl, and SO,Cl at 70-80°c., and the whole heated for several hr. at 80-85°c.

Polybrominated Dibenzanthrones and isoDibenzanthrones. General Aniline.

Dibenzanthrone and isodibenzanthrone are brominated in an AlCla-NaCl melt containing a tertiary base, e.g. pyridine, a halogen carrier, e.g. FeCl<sub>3</sub>, and during the final stages an oxidising agent, e.g. NaNO<sub>3</sub>, to give tri- to pentabromo derivatives containing < 1% Cl<sub>2</sub>. Thus, dibenzanthrone (46 g.) is dissolved in a mixture of AlCl<sub>3</sub> (300 g.), NaCl (45 g.) and pyridine (30 c.c.) at 100°c. After cooling to 80-90°c., iodine (2 g.) is added followed by bromine (37 g.) over 1 hr. Heating at 90°c. is continued for a further 1 hr. followed by 2 hr. at 125°c., NaNO2 (12 g.) added over 2 hr. and the temperature raised to 140°c. Tribromodibenzanthrone is obtained by diluting with water and filtering.

Polyamino Dibenzanthrones and iso Dibenzanthrones. BP 709,400

General Aniline. Tri- to penta-bromodibenzanthrones and similar bromoisodibenzanthrones, prepared by the method of BP 708,752 (above) or in chlorosulphonic acid, are converted, by heating under pressure with aq. NH2 in presence of a Cu salt, to the corresponding polyamino-derivatives, which are grey to black vat dyes, fast to light and Cl<sub>2</sub>. Thus, the product obtained as described in the preceding abstract, is heated at 200°c. for 6 hr. with 27–35% aq. NH<sub>3</sub> containing CuSO<sub>4</sub>. R. K. F.

4-Hydroxy-7:8-phthaloylcinnolines. Basf. BP 709,614 1 Amino-2 sectylanthraquinone and its substituted derivatives are treated with HNO<sub>2</sub> in aq. H<sub>2</sub>SO<sub>4</sub> to produce 4-hydroxy-7:8-phthaloylcinnolines which are intermediates for dyes. Thus, all parts being by weight, 1-amino-2-acetylanthraquinone (26-5) is dissolved in H<sub>2</sub>SO<sub>4</sub> (200), water (100) added with cooling, followed by a suspension of NaNO<sub>2</sub> (8.6) in H<sub>2</sub>SO<sub>4</sub> (100). The mixture is stirred at 10°c, for 1 hr. and poured into water (1200) and ice (400). After screening off the black ppt, the filtrate is boiled to complete the cyclisation, cooled and the product (27·2)—

filtered off.

R. K. F.

β-Form Metal-free Phthalocyanine. Ciba.

Metal-free phthalocyanine in the  $\beta$ -form is obtained by heating an alkali metal phthalocyanine (at. wt. of metal > 20) with an NH  $_4$  salt of an acid of dissociation constant < 10 $^{-4}$ . A solvent may be present. Thus, sodium phthaloeyanine is boiled for 5 hr. in trichlorobenzene containing NH,CL R. K. F.

Non-crystallising Copper Phthalocyanine. BP 707,601 Aniline.

Copper phthalocyanine is heated at 140-180°C. in a mixture of AlCl<sub>2</sub> and NaCl to give on diluting in water, a pigment which does not crystallise in presence of organic solvents, e.g. toluene. R. K. F.

Phthalocyanines, Mixtures of Alkali Metal and Metalfree. Ciba.

Mixture of alkali metal and metal-free phthalocyanines are obtained by treating a solution of phthalonitrile in an alcohol, which may be diluted with an inert organic solvent, with an alkali metal. Thus, sodium is added slowly to phthalonitrile dissolved in a mixture of decahydronaphthalene and benzyl alcohol at ca. 130°c., and the whole finally heated at 180°c. for 40 min. R. K. F.

Phthalocyanine-Anthraquinone Leuco Sulphuric

Esters. FBy.

Phthalocyanine derivatives containing at least two SO₂Cl groups and a total of > four SO₂Cl and SO₂H groups are condensed with 1 mol. per 80,01 of a leuco sulphuric ester of an anthraquinone containing I NH, group. The resulting sulphonamide is alkylated to attach an alkyl having > 4 C atoms to each sulphonamide N. The products are blue-green leuco-sulphuric ester dyes. Thus copper phthalocyanine-4:4':4":4"'-tetrasulphonic acid is treated with SO<sub>3</sub>Cl in ClSO<sub>3</sub>H at 80-90°C. for 1-3 hr. The resulting tetrasulphonhoride is stirred with an aq. suspension of β-methylamine-anthraquinone-leuco-sulphuric ester in the presence of Ba<sub>2</sub>CO<sub>3</sub> for 12 hr. at 10°c. The product is separated by adding ethanol. R. K. F.

Acid Milling of Vat Dyes. General Aniline. BP 710,229
Vat dyes are produced in very fine particle size by milling 1 part of dye with 0.5-5-0 parts of a strong, normally liquid, non-oxidising acid and drowning the resulting thick magma in water. The apparatus used to mill the mixture must exert a uniform shearing action throughout the mass.

Leuco Vat Dyes. Basf. BP 707,681
The free leuco compounds (vat acids) of vat dyes are BP 707,681

repared by treating a dispersion of the dye with sodium hydrosulphite in presence of an alcohol of < 6C. Thus, Jade Green is dispersed in 1% aqueous N-methylpyrrolidone and sodium hydrosulphite added at 75-80°c. The resulting suspension may be used directly for dyeing by preliminary padding; or the vat acid may be filtered off and converted with a dispersing agent to a powder. R. K. F.

Monomethin Dyes containing a Thiobarbituric Acid Nucleus. Kodak. BP 710,846 Dyes of formula-

(R<sup>1</sup> and R<sup>2</sup> = Alk, cyclo Alk, Ar or aralkyl; R<sup>3</sup> =  $C_nH_{2n-1}$  (n = 1-4); Z = atoms to complete a heterocyclic nucleus of the pyrrole series) have high absorption properties and are readily bleached in photographic bleaching baths.

They are prepared by condensing a suitable aldehyde with a substituted thiobarbituric acid.

C. O. C.

Blue Ceramic Pigment. Ferro Corpn. USP 2,644,767 A blue ceramic pigment has its major pigmentation provided by compounds of Co in combination with compounds of Al, Ca, Zn or Si, increased pigmenting value being obtained by adding small amounts of compounds of molybdenum. The preferred proportions are black cobalt oxide (20-40 parts by wt.),  $Al_2O_3$  (40-70), ZnO (1-5),  $SiO_3$  (1-5) and  $Mo_3O_3$  (0-5-10-0). C. O. C. Solid Caramel of High Tinctorial Power. Union Starch & Refining Co.

Sugar is burnt to a tinctorial power of 20-50 (preferably

closer to the upper limit) and the hot mass discharged on to closer to the upper limit) and the not lines about, after a chilling and breaking mechanism. The product, after grinding, has a tinetorial power of approx. 45 and 4.5% of C. O. C.

Crystalline Silicon as a Black Pigment for Vitreous Enamels (V this page).

Water-soluble Azoic Cou Dyeing (VIII p. 373). Azoie Coupling Components for Wool

Existence of a >C(-O-)<sub>2</sub> Chromoph Absorption of Cellulose (XI p. 377). Chromophore - Ultraviolet

#### V-PAINTS; ENAMELS; INKS

Loss of Drying on Ageing—Properties of Hydrate Lakes. D. M. Nace and W. C. Walker. Ind. Eng. Chem., 46, 769-773 (April 1954).

The following factors were found to cause or contribute to the loss of drying on ageing of printing inks containing alumina hydrate lakes—(1) high acidity of the pigment (2) presence of moisture (3) presence of Turkey red oil (4) high concentration of dye in the lake. The loss is due to adsorption of cobalt from the drier on to the pigment. Two adsorption mechanisms are suggested—(a) extraction of cobait into the acidic water film surrounding the pigment (b) reaction of the cobalt with oil soluble sulphonic acids derived from Turkey red oil and incompletely fixed dyes. A cobalt adsorption test, to distinguish those alumina hydrate lakes liable to cause loss of drying on ageing, is W. K. R. described.

#### PATENTS

Black Dye Composition for Use in Hectograph Inks.

General Aniline. Addition of a large proportion of a spirit-soluble nigrosine to the shading dyes used in a black hectograph ink yields a product which produces a good black in many more copies without deterioration to one of the component colours. Probably as the shading colours become exhausted the nigrosine asserts its influence, preventing domination by any of the shading dyes and maintaining the black throughout.

BP 710,116 A black hectographic dye composition which remains A black and strong after repeated use is obtained by mixing an azo dye of formula  $(H_2N)_R$ -RXY.N:N-RXYY.Z (n=0,1 or 2; R and R' = same or different Ar; X and Y = same or different, H, Hal, Alk or alkoxy; X' and Y' = same or different, H, Alk, alkoxy or carboxy; Z = H, NH<sub>2</sub>, Alk, between the last constant of the constant of th

hydroxyalkyl or acidyl) with a shading dye, e.g. Euchrysine or Crystal Violet and blending this mixture with soluble dye salts which combine to produce a black.

Aminoaldehyde Condensates as Printing Inks and Paints. Catalin. BP 710.171

An ink which yields non-tacky films consists of a dispersion in a non-aqueous liquid of a non-resinous etherified aminoplast precondensate, a catalyst containing at least one amino group in the aminoplast and, as drier, an acid or acid-forming substance which if the product is intended as a printing ink, does not corrode zine, or in the case of a paint does not corrode the surface to be painted, together with pigments, plasticisers, solvents, etc. C. O. C.

Printing Ink. Sterling Drug. USP 2,644,759-60 Addition of a small amount of the wetting agent obtained by condensing a polyethylenepolyamine with urea or propylene oxide imparts to printing inks stable viscosity, improved flow characteristics and resistance to bleeding.

Steam-setting Printing Inks. Sun Chemical Corpn USP 2,645,622

Inks yielding glossy prints contain as vehicle a waterinsoluble resinous binder dissolved in a polyglycol solvent, there being also present an alcohol-soluble N-alkoxymethylpolymethylenepolyamide of the type described in USP 2,430,860.

Polychlorotrifluoroethylene British Coatings. BP 702,911 Thomson-Houston Co.

Coatings of polychlorotrifluoroethylene are applied, e.g. to wire, from a fine dispersion of the solid polymer in a mixture of a liquid polymer of chlorotrifluoroethylene with another compound, e.g. a-fluoronaphthalene, which together dissolve the solid polymer on heating with, as a diluent, an alcohol, ether, ketone, ester etc., e.g. butanol. The applied coatings are consolidated and solvents recovered by baking at 350-425°c.

Surface Coating Compositions. Plastanol.

BP 710,441 A coating composition which yields a hard water-A coating composition which yields a man vac-resistant film and is suitable for use on porous surfaces, consists of an aqueous emulsion of an organic solvent solution of an isomerised rubber resin, e.g. of the type described in BP 498,311, 539, 641 and 541,899 or those prepared by treating rubber with chlorostannic acid. C. O. C.

Crystalline Silicon as a Black Pigment for Vitreous Enamels. British Thomson-Houston Co.

BP 710,064 A dark grey vitreous enamel consists of a frit of SiO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, ZnO and as pigment 5-20% (on the wt. of frit) of crystalline silicon. crystalline silicon.

#### VI—FIBRES; YARNS; FABRICS

Cellulose Chemistry and the Textile Industry. D. A. Clibbens. J. Textile Inst., 45, P173-P193 (May 1954).

Freezing of Swollen Cellulose Fibres. N. I. Klenkova. J. Appl. Chem. U.S.S.R., 27, 433-444 (April 1954). When wet cellulose fibres are carried through a series of freezing cycles, the amount of non-freezing water (cf. J.s.D.c., 70, 315 (July 1954)) in the fibres may fall when

the amount of water present is not great, but will otherwise rise progressively, eventually attaining very high values (there is a parallel increase in the swelling in caustic alkali solutions). Progressive increase in content of non-freezing water is observed also in similar experiments on chitin, ethyl cellulose, and lignin. It is found also that finely ground cellulose pulps undergo considerable aggregation during the first freezing cycle, and are then progressively broken down in further cycles. Analogous phenomena are observed in starch solutions—the first freezing results in irreversible precipitation of starch of lower hydrophilic character, but in further cycles there is a progressive increase in content of non-freezing water. It is considered that two opposing processes occur during freezing—de-hydration and compacting of the macromolecular system due to passage of water from between the chains to crystallisation centres, and opening of the structure due to the splitting effect of growing ice crystals.

Bubble Pressure Test. II—Application to a Study of the Sealing of Close Cotton Cloths when Wetted. J. Lord. J. Textile Inst., 45, 7380-7389 (May 1954).

Using the bubble-pressure test, the sealing due to the swelling of close cotton cloths when increasing amounts of moisture are present is followed. Effects of waterproofing agents and of different cloth structures are studied and discussed. J. W. B.

Elongation of Fibres under Stress. W. Wegener. Melliand Textilber., 35, 40-44 (Jan.), 156-157 (Feb. W. Wegener. 1954).

The elongation and change in elongation as functions of time and stress of Perlon, four rayons, and both raw and boiled-off silk and tussah are examined under conditions of stress increasing uniformly with time.

Mercerisation of Acid-treated Jute Fibre. 8. M. Mukherjee. J. Textile Inst., 45, T 405-T 411 (May 1954). Jute is pretreated in excess H<sub>a</sub>SO<sub>4</sub> of various concentrations at 20°c. for 48 hr., washed, dried, and treated in 20% NaOH at 20°c. for 2 hr., a period known to be more than sufficient to complete the process of mercerisation in pure cellulosic fibres. X-Ray photographs show that the proportion of the hydrate modification progressively increases with increase in acid concentration, being practically complete when the concentration is 500 g./litre. Under these conditions the fibres become very weak and brittle. Ramie and jute are also treated overnight in 65° HNO, at room temp. and X-ray photographs taken of the acid-soaked specimens. Characteristic patterns of the Knecht compound are obtained. After washing and drying, almost complete hydrate transformation is found in jute,

J. W. B. Influence of Retting on the Physicochemical Properties of Hemp. G. Centola. Teintex, 19, 241-253 (April 1954).

characteristic of nitrated cellulose. Results are discus

and an appreciable amount of native cellulose in ramie. Treatment of ramie with 70% HNO<sub>3</sub> leads to patterns

Microbiological retting is more thorough, but less controllable, than chemical retting. The ill defined compounds known as pectins, lignins, and hemicelluloses may be present in physical admixture, or in chemical combination, with hemp cellulose and are subject to wide variations in type and amount. Such variations can profoundly affect a fermentation process. The location of pectin in green and fermented hemp was studied by staining the solvent-extracted fibres with the carbinol base of Crystal Violet. In green hemp, the pectin was found to be present in planes on the exterior of the fibres, with marked concentrations in certain zones transverse to the orientation of the fibrils. Retted bemp showed the pectin to be located at fairly regular intervals along the fibre, the elementary fibres being linked together in those areas and free elsewhere. Two types of polyuronide macromolecules, of different resistance to micro-organisms, are postulated to explain the residual, resistant pectin bridging the fibrils,

by analogy with the lignin of jute.

Chlorite bleaching of hemp at pH 4-0-4-5 and 65°c. was found to be ineffective unless carried out in two stages with an intermediate alkaline treatment. From this it is deduced that lignin exerts a protective action on pectin, the two being present in hemp in intimate association

8. R. C.

Machines for Preparing Sisal, F. Mertz. Melliand Textilber., 35, 202-204 (Feb. 1954).

New Aspects of the Structure of Wool. E. H. Mercer.

J. Textile Inst., 45, T 365-T 367 (April 1954). Evidence is presented which suggests that apparently asymmetrical behaviour of the wool fibre is not due to the cuticle. Fibres are damaged by alcoholic alkali,  $Br_{\parallel}$  in  $CCl_4$ , and by buffered trypsin; subsequent dyeing shows a marked ortho-para differential staining. This staining is repeated on cortical cells from wool given a prolonged and complete enzymic treatment, and about half the cells show Similarly, in 5x-NaOH about half greater staining. ontract more rapidly. It is suggested that this ortho-para differentiation is due to uneven keratinisation during fibre growth, and that the ratio within the fibre may have an important influence on the handle of fleece wool and also on subsequent behaviour such as felting and tangling. J. W. B

Reversible Twist and Bending Phenomena in Keratin Fibres. G. King and F. L. Warburton. J. Textile Inst., 45, T 412-T 418 (May 1954).

Experiments have shown that, although if wool yarns with a tendency to snarl are wound on bobbins and wetted some of the tendency is removed, a far greater removal is effected during subsequent drying. The effect is reversible, a snarl reappearing on wetting. Studies are accordingly made on cow-tail fibre, and to a smaller extent on nylon and rubber monofils. A single fibre and torsion system, enclosed to permit regain control, is used, and the effect of swelling after sorption of formic acid is also studied. It is shown that, for equilibrium elastic constants, the reduction in modulus is so small that the effect of radial swelling predominates and the fibres show increased stiffness with J. W. B. increased regain.

Relaxation Shrinkage of Woven Wool Fabrics during

Soaking in Water and Hoffman Pressing. A. N. Davidson. J. Testile Inst., 45, T 323-T 347 (April 1954). Relaxation properties of a wide range of woollen and worsted woven fabrics are examined by soaking in water, in solutions of different pH values, and by Hoffman pressing and steam treatment. Measurements are made after conditioning in a standard atmosphere, as wet measurement cannot be made. Among the many results presented the following are the most important factors in determining the extent of shrinkage: temperature and pH; the number of soaks; the relative humidity of the conditioning prior to measurement; and the specimen size. Shrinkage during pressing is greater if the steam is wet, and London shrinkage does not always render a fabric stable to Hoffman pressing.

In appendix (a) the effect of sett on shrinkage is studied, and in appendix (b) (by A. N. Davidson and B. F. J. Moxon) curved- and flat-bed presses are compared. Tightly woven fabrics are more stable than those with an open weave and the differences in shrinkage obtained with the two types of press are small, the curved-bed giving slightly higher values than the flat-bed. Application of vacuum with the head unlocked tends to give a greater shrinkage than with it J. W. B.

Structure of Silk. H. Zahn and H. Zuber. Textil-Rund., 9, 119-127 (March 1954): Teintex, 19, 350-367 (May 1954).

Three electron-micrographs illustrate that macerated fibroin consists of fibrils which normally lie parallel in bands but may be torn apart. When cross-linked with pp'-diffuoro-mm'-dinitrodiphenyl sulphone (I), the bands cannot be separated into fibrils by maceration but only into irregular fragments. This suggests that some of the cross-linking is inter-fibrillar, and that fibroin consists of concentric layers of fibrils. It is possible that weighting takes place as a deposition between these layers. Only of the tyrosine in fibroin is accessible to 2:4-dinitrofluorobenzene and none of the other cpd. investigated reacted to a greater extent. Apart from reacting with the e-amino groups of lysine, I reacts bifunctionally with the accessible tyrosine in fibroin, which must therefore be spaced ca. 10 a. apart. This implies that along each molecule at most every sixth residue is tyrosine, but as fibroin contains only enough tyrosine for ca. one residue in twenty, the tyrosine residues in fibroin must be unevenly distributed along the length of the molecules. X-Ray diagrams show that the crystalline portion of fibroin is unaltered on cross-linking

Fibroin cross-linked with I is less reactive towards HCl, LiBr, and ethylenediamine soln., a contraction of ca. 60% occurring instead of dissolution. The supercontracted form shows a disoriented X-ray diagram, but stretching reestablishes the fibrous structure. Fibroin cross-linked with I on hydrolysis (HCl, 120 hr., 22-24°c.) can be divided into a sol. and an insol. portion. The latter, as expected, contains the large fractions bound to I, which consist of glycine, alanine, serine, and glutamic and aspartic acids (each ca. 9-10% by wt.) and tyrosine (35-45%), whereas the former contains very much glycine and alanine, and much serine, though tyrosine and valine are also present. Assuming that the insol. portion corresponds to the amorphous part of fibroin, this amounts to about 25% of the silk. From this work, it follows that in the amorphous regions every fifth aminoacid residue is tyrosine. suggested that every chain-molecule of fibroin consists of about 30 crystalline and amorphous segments. 4-Fluoro-3nitrobenzenesulphonic acid alkylates the hydroxyl group of tyrosine, the  $\epsilon$ -amino group of lysine and the amino end groups of silk. Using this reaction, the lysine content was found to be 0.67%, and the end-groups 0.10-0.14%, i.e. mol. wt. of 80,000-100,000. The end-groups are due probably to glycine and alanine and some other amino acids. The reaction can also be used for the colorimetric determination of amino groups (found 1.05 millimole/10 g.). similar approach is being employed in the case of collagen.

Ultraviolet Absorption Spectra of Silk Fibroin and Cellulose in Lithium Bromide Solution. E. Schauenstein, E. Treiber, W. Berndt, W. Felbinger, and H. Zima. Monatsh. Chem., 85, 120-139 (Feb. 1954).

The dissolution of pure degummed silk fibroin in conc. LiBr leads to a decrease of peptenolate absorption in the region of 4000 mm.<sup>-1</sup>, and to a displacement of the phenolic tyrosine dissociation curve towards the lower pH-values; this indicates a rupture of hydrogen bonds between the peptide groups. The effects are shown to be reversible from measurements with dialysed silk solutions. Analogous measurements with cellulose support current views on the mechanism of photolysis, and also enable certain deductions to be made on the nature of  $\beta$ -cellulose. H. H. H.

Processing of Synthetic Fibres and Yarns. Forschungs-kuratorium Gesamttextil. Melliand Textilber., 35, 1-2 (Jan. 1954).

A brief, preliminary report is given on a research scheme expected to cost DM. 500,000 and to be tackled jointly by the research institutes of Western Germany. hand includes studies in carding, spinning, properties of mixtures with other fibres, heat setting, dyeing under pressure (speed of uptake of dye, choice of dye, alterations in degree of dispersion of dyes, changes of colour, improved cuprous ion technique), yellowing of fluorescent whitening agents, washing, removal of static, and the viscosity of Perlon.

Phrilon and Redon. J. Wrieth. Textil-Rund., 9, 83-84 (Feb. 1954)

Both synthetic fibres are made by Phrix-AG, Hamburg. The former is made from ε-aminocaproic acid, the latter is a polyacrylonitrile. Some of their properties are, respectively: density, 1·13, 1·14; strength, 40-50, 25-30 Rkm.; extension at break, 40-50, 30-40%; regain at 65% R.H., 20°c., 4, 1·3%. The melting point of Phrilon is 213-215°c., but 140°c. should not be exceeded in ironing, nor 165°c. for fabrics made of mixtures containing Phrilon. Redon, which is cream-coloured, becomes pale brown above 125°c. dark brown at 180°c., but maintains 60% of its strength even after 16 days at 150°c. It should not be ironed above Other properties, possible uses, and qualities ctured are also given. H. E. N. manufactured are also given.

Dynel "Inner Structure". T. A. Feild, C. A. Setterstrom, and C. W. Davisson. Amer. Dyestuff Rep., 43, P296– P303 (10 May 1954).

When the rayon is dissolved from a stock-blended rayondynel fabric, what is known as the dynel "inner structure" is disclosed. It is suggested that such structures are extremely useful in determining the influence of fibre components on dimensional stability, colour, light fastness, uniformity of blending, heat and abrasion damage, loft, and

pleat retention, of the original fabric. Methods are described for preparing the structures, and examples are given of their use in determining fabric behaviour.

Movil. G. C. Zuccari. Melliand Textilber., 35, 9 (Jan. 1954). An Italian polyvinyl chloride fibre marketed as filament and staple. It has sp. gr. 1.37, does not support combustion, weakens at  $70^{\circ}$ C., and remains flexible at  $-80^{\circ}$ C., becoming  $50^{\circ}$ 6 stronger. It shrinks at  $65^{\circ}$ C. in water or at  $75^{\circ}$ C. dry and can therefore be employed for special effects.

### PATENTS

Prevention of Milkiness in Viscose Rayon. Algemene Addition to the viscose solution of small amounts of a soluble cation-active compound of formula

 $(CnH_{2n}O)_xH-RN-(CnH_{2n}O)_yH$ (R = aliphatic hydrocarbon of > 8C; n=2,3, or 4; x+y>5)

e.g. Ethomeen C/25 (Armour & Co.) serves both the purpose of inhibiting milkiness in the rayon yarn and reducing spinneret incrustation in the spinning bath.

Dry Spinning of Viscose. Textile & Chemical Research BP 709,629 Regenerated cellulose fibres are produced by extruding a dispersion of cellulose xanthate in caustic soda having

 $\eta>1,000$  poises, cellulose content 6% and a caustic soda content of 3-10% into air at room temperature, stretching the product and treating with cold mineral acid. BP 709,700

An improved method of producing the xanthate dispersion is described in which the xanthate is repeatedly passed between calendering rolls at < 10°c. and at the same time adding water to give the desired viscosity. W. G. C.

Crimped Textile Yarn. Heberlein & Co. A crimped textile yarn, made by imparting a high degree of temporary twist to continuous filament yarn of wholly synthetic organic material, particularly polyamides, by steam-setting the temporary twist, and untwisting or even reverse twisting, is improved by stretching by 10-70% and steaming in the stretched state for up to 30 min. at 1-3atmos. pressure. Stretching is done in skeins on uniformly curved surfaces, or by an expanding reed comprising a plurality of curved surfaces, or by placing the skein on parallel rotating rollers which are then drawn apart. I. W. B.

Improving the Strength of Artificial Silk Threads, Yarns, and Cords. Vereinigte Glanzstoff-Fabriken. BP 710,708

The permanent elongation is reduced and the strength improved of artificial silk threads, yarns, or cords, e.g. for use as tyre cords, by submitting the finished thread, while continuously travelling, to a rhythmically alternating tensile stress of 10-50% of the maximum, so as to apply at least 100 alternations to each elementary length of the thread, e.g. 500/min. J. W. B.

Highly Porous Absorbent Fabric-like or Chamois-leather-like Flexible Sheet Material. Carl BP 710,082 Freudenberg.

Highly porous material 0-15-0-90 mm, thick is made by impregnating in 2 stages a fleece of randomly-arranged fibres with a film-forming vulcanisable adhesive, e.g. aqueous dispersion or emulsion of natural and/or synthetic rubber. In the first stage only part of the total impregnant needed is introduced and, after drying by heating, the intermediate product is re-impregnated and dried at least once more. At some stage—either between or during the subsequent impregnations—finely-divided water-soluble pore formers, e.g. inorganic salts such as NaCl, MgSO4 or Na<sub>2</sub>SO<sub>4</sub>, or organic compounds such as sugar, starch, cellulose derivatives or albumin, are introduced. They are removed after final vulcanisation. J. W. B.

Cross-linking Reagents for Wool and their Physiological Effects (X p. 375).

Microbe-resistant Wool by Chemical Modification. Paper-chromatographic Examination (X p. 376).

### VII - DESIZING; SCOURING; CARBONISING; BLEACHING

Wet Processing of Recovered Wool. R. Siegmund.

Textil-Praxis, 9, 473-475 (May 1954).

The types of pulling oil used are mineral oils, either as such or compounded, emulsifiable mineral oils, and oleines. Petroleum oils introduce difficulties in scouring, especially since the content is high. The detergents available are reviewed, and simple tests for suitability are quoted. A practical example is given of the milling and scouring of pieces composed of 70% reclaimed wool, 20% clipped wool, and 10% cellulosic fibre. 8. R. C.

Scouring of Perlon Hose. G. Schug. Melliand Textilber., 35, 45-47 (Jan. 1954).

Six recipes for removing oil and metal stains are given H. E. N. and discussed.

Behaviour of Fluorescent Whitening Agents on Cellulosic Materials. 8. N. Glarum and S. E. Penner. Amer. Dyestuff Rep., 43, P310-P314 (10 May 1954). Some fundamental aspects of the behaviour of fluorescent whitening agents ("optical bleaching agents") on colludescent discovered in the second colludescent discovered colludes cellulose are discussed. The nature of the relation between concentration of agent and the fluorescence intensity of the system is consistent with certain empirical and theoretical laws of fluorescence and is useful for investigating the relative strengths, purities, and efficiencies, of comparable, optically active, products. Adsorption isotherms resulting from measurements of equilibria prevailing after exhaust application of "optical bleaches" are shown to be similar to those obtained with substantive dyes. Relations between fluorescence intensity and visual whiteners are also considered, and the value and limitations of fluorescence measurements as a measure of visual whiteness are shown.

PATENT

Washing Yarns and Fabrics. Dutton and Reinisch. BP 709,557

Yarn and fabric is treated with an emulsion of a soapforming fatty acid or acids, containing also 1 or more soapcompatible sulphated acyl alkanolamides, and then with a solution of alkali to generate soap in situ. J. W. B

### VIII— DYEING

Matching Colours. M. Richter. Textil-Rund., 9, 141-147

Reprinted from Die Farbe, 1, No. 2 (Oct. 1952). The relationship between the light used, the samples, and the observer is concisely explained.

Heat Retention and Skin Irritation of Dyeings. F. Weber. Öster. Textilztschr., 7, 212 (1953): Textil-Rund., 9, 154 (March 1954).

Not only does heat retention depend on how dark a dyeing is, but also on the class of dyes employed in producing it. Azoic, disperse, and direct dyes reflect about 85% of the incident heat. This factor is usually lower for vat, chrome, and acid dyes and is much more variable. Dyeings of sulphur dyes reflect far less heat, i.e. they absorb the heat and become warmer (e.g. a direct black reflects ca. 70%; sulphur black 3-7% of the incident heat). The best reflection is obtained with metallic pigments. Applied to linings, they reflect the external heat in summer and therefore cool, in winter they reflect the body heat and therefore warm. Eczemas have frequently been developed by wearers of nylon and other synthetic fibres. They are attributed to: low absorbency of nylon, i.e. friction in presence of perspiration; sexivalent chromium left in chrome dyeings; certain disperse dyes; and swelling agents, such as chlorinated benzenes and tetralin, but not benzoic or salicylic acid. Properly prepared formaldehyde resin finishes are completely harmiess. H. E. N.

Azoic Dyeing. Jellinek. Z. ges. Text. Ind., 54, 823 (1952): Textil-Rund., 9, 98-99 (Feb. 1954).

Most red azoic dyeings have limited fastness to H<sub>2</sub>O<sub>2</sub> Good in this respect is Fast Bordeaux OL Salt (particularly with Naphtols AS-BO and AS-BI). Fast Navy Blue RA Salt (particularly with AS-ITR, AS-BO, AS-WS, AS-LC, and AS-S) gives navies of very good fastness to light and chlorine, but is applied from a bath containing much acetic acid. The same method is used with Fast Brown VA Salt (V brand for application by padding, jigger, or printing), which gives very fast dark browns (particularly with AS-RL and AS-VL). Difficulties experienced with raincoat-poplins dyed with azoics are discussed and attention is drawn to special ranges of azoic dyes- Ofna-lan for wool and rayon staple, Ofna-cet for cellulose acetate and polyamide fibres, H. E. N. and Rauna for furs.

Water-soluble Azoic Coupling Components for Wool
Dyeing, H. Rath and E. Feens, Melliand Textilber.,
35, 267-269 (March 1954).
The methods of obtaining water-soluble deriv. of

naphthols (I) suitable for dyeing wool and related fibres and able to couple with diazo cpd. are reviewed. Potassium salts of the sulphuric esters of I are prepared by adding chlorosulphonic acid to a suspension of I in dimethylaniling, the mixture being finally warmed to 50-60°c. On cooling, KOH is added when the dimethylaniline separates, salts are excellently sol. in hot water and are applied to wool from a boiling bath, to which is added 5% acetic or 3% formic acid. After 20 min., 10 c.c. of 10%  $H_880_4$  is added per litre, and boiling continued for 10 min. The wool is mangled, rinsed in dil. ammonia, and developed. The naphthols tend to crystallise and then to couple incompletebut this can be hindered, e.g. by addition of formaldehyde. This trouble does not occur with silk, polyamide, or cellulose acetate fibres.

Dyeing Textile Fibres under Static Pressure. Drijvers. Amer. Dyestuff Rep., 43, 243-246 (12 April 1954).

Results obtained by the Steverlynck static process in a pressure-circulating dyeing machine are described. It is claimed that dyeing can be carried out above 100°c.; that the pressure has a stabilising influence on dye and bleach baths; and that surface oxidation in reducing baths is eliminated, thus saving sodium hydrosulphite. J. W. B.

Phthalocyanines in Dyeing and Printing. A. Schmitz.

Melliand Textilber., 35, 274-276 (March 1954). A brief review. Phthalogen Brilliant Blue EF3G (FByprobably IF3G intended) requires even drying and can be used for testing the efficiency of drying machines in this H. E. N.

H. Toepffer. Dyeing of Polyacrylonitrile Fibres. Melliand Textilber., 35, 47–52 (Jan. 1954). The development, properties, and advantages of poly-

acrylonitrile fibres are described. The application of disperse, basic (particularly Astrazone (IG) dyes), acid (by the cuprous ion technique), vat (preferably indigoid), and exidation dyes and pigments is briefly reviewed, as is the dyeing of mixtures containing polyacrylonitrile fibres. For pieces, a winch machine, modified by inserting additional steam coils, lowering of the winch, and having a tightly fitting cover under which steam can be injected, enables medium dyeings on filament and heavy dyeings on staple to be achieved. A laboratory apparatus for dyeing yarn under pressure is illustrated by five photographs. H. E. N.

Dyeing and Finishing Polyvinyl Chloride Fibres-New Developments. 70-76 (Feb. 1954). L. Gord. Textil-Rund., 9,

Two types of polyvinyl chloride fibres are considereddrawn, crystalline staple fibres or filaments which shrink above 75°C., and thermally stabilised amorphous staple The latter absorb dyes more readily. of dyes are applied in the presence of suitable surface-

Vat dyes are dyed from a bath neutralised with acetic acid and give a comparatively good yield, but insufficient rubbing fastness. Solubilised vat dyes are commercially important, difficulties being obviated by addition of a cellulose bisulphite dispersant. Solid shades can be cellulose bisulphite dispersant. obtained on unions with nylon by padding. With sulphur dyes, dull shades of good fastness to rubbing and washing, but poor to light, can be obtained. A great number of azoic combinations can be used, particularly on stabilised fibres, but in some cases the fastness to light is low. Lists are given of those disperse dyes which give good yields. The variation of yield with temp.  $(85, 90, \text{ and } 100^{\circ}\text{c.})$  is given for three dyes (1%, 1 hr.) and, as the yield greatly depends on temp., dyeing is carried out as near the boil as possible. The variation of yield for the same three dyes with amount of dye (1-10%; I hr., 100°c.) and with time (30 min.-2 hr.; 3% dye, 100°c.) is also recorded. Increases in yield lag far

behind increases in the amount of dye used and time has little effect. Though not all are suitable, disperse dyes are most used as a class and have good fastness to rubbing, chlorine, sea-water, and perspiration, and to washing  $<70^\circ\mathrm{C}.>70^\circ\mathrm{C}.$ , migration and bleeding occurs. Light fastness is up to 5–6, but once light begins to affect these dyes, fading occurs very rapidly. This is shown not to be due to decomp. of the fibre. Latyl (DuP) brand dyes have very good fastness on polyvinyl chloride. Stripping is difficult, acid hypochlorite at the boil or Na chlorite in presence of  $\mathrm{H_2PO_4}$  or  $\mathrm{HNO_2}$  being best. Pigments are fixed after drying at 65–70°C. by a passage through NaOH and give good light fastness, but the rubbing fastness is good only in pale shades. Polyvinyl chloride always becomes negatively charged, the maximum charge being attained immediately and persisting for a long time. Only one antistatic agent (DuP) proved resistant to a number of washes, but admixture of 10-20% of another fibre, e.g. a cellulosic one, is a satisfactory alternative. Crease-resisting is unnecessary. Stiffness, e.g. for filter cloths, is obtained by a heat treatment at 85–90°c, without allowing shrinkage to take place. By means of a treatment in a hot bath, mixtures of polyvinyl chloride and non-shrinking fibres can give imitation buckskin.

Survey of Colouring Matters used in Foods. E. V. Edding. Fac. quim. y farm., Tesis quim. farm., 3, 447-478 (1951).

PATENTS

### Colouring Textiles of All Types with Vat Dyes. Basf. BP 709,150

Textiles of all types when impregnated with aqueous dispersions of water-insoluble vat dyes in which the dye particles are  $<10m\mu$  in size and then dried are coloured as if the dye had been applied either by dyeing or printing by one of the customary reduction and reoxidation methods. The dye particles because of their very great thermodynamic spontaneous movement penetrate into the internicellar spaces of the fibres where they are retained particularly after drying. 
C. O. C.

Azoic Dyeing of Acetate Rayon. Celanese Corpn. of America. USP 2,643,175

Improved azoic dyeings are obtained on acetate rayon if the acidity is kept low during diazotisation, e.g. by use of 0-3-0-5% of acetic acid in the diazotising bath, and after the dye has been developed treating the material with a swelling agent, e.g. in 20-30% aq ethyl alcohol at 20-30°C, for 1-3 min. The resultant dyeings have increased fastness to light and improved penetration.

C. O. C.

Dyeing Nylon with Acid Dyes. ICI. BP 710,103
Addition to the dyebath of a salt of a naphthalene di- or trisulphonic acid results in more level dyeing. C. O. C.

Reserving Nylon against Acid and Mordant Dyes when mixed with Wool. Gy. BP 709,977 Polychlorodiphenylurea or thiourea monosulphonic acids of formula—

(X = O or S; Y = H or Cl) are very efficient resisting agents, 0.5-5.09% giving complete resistance to acid or chrome dyes without affecting the dyeing properties of wool mixed with the treated nylon. C. O. C.

Dyeing Aromatic Polyesters. ICI. BP 710,303
Good dyeings are obtained by treating aromatic polyesters with the acid leuce compounds of a vat dye in presence of a swelling agent, e.g. a phenol. C. O. C.

Hair Dye Composition. Union Française Commerciale et Industrielle. BP 710,

Hair can be dyed from blonde to reddish brown bluish black in absence of metallic salts by use of a mix of a compound of formula—

and a sulphite, hydrosulphite or mercaptan of formula SH·R·Y (R = a bivalent aliphatic residue which may contain an alcoholic group; Y = H or COOH), adding to this mixture immediately before application to the hair, ammonia or a primary, secondary or tertiary aliphatic or cycloaliphatic organic base or a salt (of basic reaction) thereof. C. O. C.

Colouring Foodstuffs. Deutsche Hoffman-La Roche.

BP 710.789

Polyphase foodstuffs in which one component is water and a material which forms a dispersion in water is another component, e.g. margarine, puddings, etc. are coloured by addition of a material which colours the continuous phase and another which colours the disperse phase, e.g. carotene or bixin is dissolved in fat and riboflavin is dissolved in water and the solutions emulsified in water-free margarine fat in the usual manner.

C. O. C.

**Hair Tinting Composition.** Charles Marchand Co. USP 2,645,595

Use of benzene phosphonic acid C<sub>5</sub>H<sub>5</sub>PO(OH)<sub>2</sub> as the acid dye fixing agent in a hair dye concentrate, prevents the concentrate absorbing water from air and renders it stable for long periods without staining or degrading the package in which it is. Thus a henna hair rinse consists of D&C Brown No. 1 (0·011 g.), Ext. D&C Red No. 13 (0·003), benzene phosphonic acid (0·500) and boric acid (1·500) this being dissolved in 1 quart of water when it is desired to use it. C. O. C.

Processing of Synthetic Fibres and Yarns (VI p. 372). Some Aspects of the Application of Dyes to Paper (XI p. 377).

Chromatographic Method for Examining the Dyeing Properties of Wool Dyes (XIV p. 380).

### IX-PRINTING

### Thickening Agents—Measurement of the Properties of Pastes. K. Schmidt, Melliand Textilber., 35, 177-183 (Feb. 1954).

To measure the clarity of prints (I) obtainable with a certain paste, it is printed using specially designed screens in which the pattern varies from very fine apertures to very coarse, e.g. a wedge or tapering spiral, and the reproduction is expressed as a percentage of that possible. The degree of penetration (II) is also given as a percentage by—

Whiteness of material — Whiteness of throughprint  $\times$  100%. Whiteness of material — Whiteness of print

Complete reversibility is therefore 100% and total lack of striking through 0%. Instruments for measuring the viscosity, surface tension, and extensibility (Zügigkeit; III) of printing pastes are described and discussed. The optimal conen. for I occurs with wheat starch (W) pastes at 80 g./kg. and for gum arabic (A) at 480 g./kg., being almost 100% in both cases. The optimal conen. for I is found to be also the optimal conen. for III, the latter being more readily determined. Optimal conen. for III occurred at the following values of III (mm.) and conen. (g./kg.)—gum tragacanth, 25, 50; W, 25, 80; Tragu 8, 50, 80; Alkagum N, 155, 165; Thickener HD (cellulose ether), 90, 200; Industrial gum, 100, 420; British gum, 85, 450; A, 100, 480. III varies with temp., exhibiting a maximum usually below 40°c. The viscosity at these conen. is about 2000 centipoises for W, A, gum tragacanth, Industrial and British gum, but higher for the others. I and II are inversely related and the best compromise lies at the following conen. (g./kg.)—Alkagum N, 33; W, 43; Tragu S, 77. H. E. N.

Crimp, Blister, Embossed, and Gloss Effects. R. Hofmeister. Melliand Textilber., 34, 1075 (Nov.), 1157–1161 (Dec. 1953); 35, 61–64 (Jan.), 174–177 (Feb. 1954).

An extensive review, with references, of the modification of the surface of textiles by printing processes. H. E. N.

Preparation of Screens for Printing. H. Hünlich.

Textil-Rund., 9, 95-97 (Feb. 1954).

A review from the practical point of view. H. E. N.

Screen Printing Today. G. Bernardy. Melliand Textilber., 34, 859-860 (Sept.), 963-964 (Oct.), 1073-1074 (Nov.), 1154-1157 (Dec. 1953); 35, 53-58 (Jan. 1954).

An extensive review, excluding matt, bronze, pigment, paint, and flock printing. Steaming in the rapid agers used

for roller printing is unsuitable because of the larger amounts of dye on the fabric and more successful machines are described. The first aftertreatment is best carried out in open width. For clean discharges with pastes containing vat dyes the following precautions are advocated: use of pastes with more body (e.g. employing British Gum), less hygroscopic agent, less alkali (Na<sub>2</sub>CO<sub>3</sub> rather than K<sub>1</sub>CO<sub>3</sub>), pretreatment of the piece with a small amount of an oxidising agent (e.g. m-nitrobenzenesulphonic acid), and steaming with somewhat drier steam. In screen printing with vat dyes by hand 20-30% more sodium formaldehyde sulphoxylate must be used, because of greater decompo-sition. Rapid drying, best with dried air, is essential, followed by quick steaming and development. The advantages of the Colloresin process are listed. For coloured discharges on azoic grounds, anthraquinone is added, but this reduces the yield in some cases, e.g. anthraquinonoid vat blues. Resists are produced using aluminium sulphate or tartaric or lactic acid, and can be modified to produce coloured resists. For Rapid Fast colours, acid or reducing thickeners, e.g. British Gum, should be avoided. Development can be carried out either by hanging overnight in damp, warm rooms or, continuously, by neutral steaming at 100°c. for 3-5 min., followed by a hot liquor containing acetic or formic acid, or by acid steaming. Rapidogen colours must be developed in acid steam or in a hot, acid bath. Rapidogen N colours (FBy) require only neutral steam. Recent developments in oxidation colours are Indosol Black T (DH) and Solanile Black (Fran), in the phthalocyanine field Phtalogen Brilliant Blue IF3G and Green IFFB (FBy) and Alcian Blue 8GS (ICI). Solution Salt B (Basf) is a useful auxiliary product in printing disperse dyes on cellulose acetate. Prints of vat dyes on cellulose acetate tend to run, hence less potash is used and the steam must not be too wet. As in screen printing more dye is transferred on to the fabric than in roller printing, trouble is experienced with nylon and Perlon and the structure of the material and the type of design are very important. The metal-complex dyes, Vialon Fast (Basf), are easily applied. Discharges on cellulose acetate, nylon, or Perlon are produced using zinc formosul. Leuco esters of vat dyes on weighted or unweighted silk are best developed by means of chromic acid. Unweighted silk is best prepadded with diethyl tartrate before printing with Rapid Fast and Rapidogen colours and then drying and steaming neutral for 5-20 min. In printing wool, not only should the steam be wet, but the material should be damp. Mordant colours have lost much of their previous importance in printing, but some are used for undischargeable contours.

H. E. N.

### PATENTS

Anchoring Deposits on Lacquer Surfaces. American Research. BP 710,643 When forming deposits on lacquer surfaces, e.g. by

vacuum evaporation of metals, distortion of the surface is avoided by firstly partly polymerising the lacquer, then depositing the metal or other material and finally completing curing of the lacquer at a temperature not below that used for the preliminary polymerisation. C. O. C.

Transfers. R. S. Colour Works. BP 710.807 Transfers. R. S. Colour Works.

The transfer material after being applied to the backing, is coated with wax, those parts of the backing having no coated with wax, those parts of the backing having no coated. This offers advantages in applying the transfer, e.g. in the case of ceramic transfers there is no need to give the article a lacquer coating before applying the transfer in order to ensure complete removal of the fired wax outside the C. O. C. decoration.

Colour Photography. T. G. Clarke. BP 709,559

Gevaert Photo-Producten and Colour Photography. hromart Film Industries. BP 710,481

Phthalocyanines in Dyeing and Printing (VIII p. 373). Colouring Textiles of All Types with Vat Dyes (VIII p. 374). Permanent Patterned Mechanical Effects on Fabrics (X p. 376).

### X—SIZING AND FINISHING

Sizes for Yarns from Glass Fibres. P. R. Masek. Melliand Textilber., 35, 226-228 (March 1954).Six recipes are examined for pH, viscosity, and pick-up

by yarn, and the sized yarns are tested for the number of breaks during winding, breaking strength, abrasion resistance, and electric resistance. H. E. N.

Superheated Steam as a Drying Medium. J. H. Hunter. Amer. Dyestuff Rep., 43, P236-P238 (12 April

When superheated steam is allowed to impinge at high speed closely and properly on to a wet fabric, improved effects over air drying are obtained. Migration of resins and dyes, and overdrying, are eliminated, the fabric is conditioned, and costs are reduced. J. W. B.

Better Mercerising for Improved Lustre. L. Fourt and A. M. Sookne. Amer. Dyestuff Rep., 43, P304-P309 (10 May 1954).

Factors influencing the lustre of cotton fabrics are reviewed, and it is suggested that the development of simple methods for measuring lustre permit improvements in mill mercerising procedures, as the individual finisher can make objective determinations of the optimum conditions of concentration, stretch, presence of wetting agents, etc. Reliance on absorption or accessibility tests, such as Ba Reliance on insorption or an instance of the misleading. It is better to use physical measurements of optical properties. J. W. B. measurements of optical properties.

Production of Felts. F. Gamba. Melliand Textilber., 35,

158-160 (Feb. 1954).
An equation is developed which explains e.g. why, in the planking of hoods by means of a multiroller machine, felts of greater density and strength are obtained by using increased pressure on rollers of smaller diameter

Shrink-resisting of Wool. O. E. I Textilber., 35, 279-281 (March 1954). O. E. Ford. Melliand

HOBr, obtained by addition of KBr to normal bleach liquors, is 10 times less dissociated than HOCl and can therefore be used at a pH one unit higher than the highest permissible with HOCl. In the two-bath Stevenson process, greater effects are produced if permanganate (I) is used before hypochlorite (II). A bisulphite treatment between I and II lessened the shrink resist. The rate of reaction of wool with I under the conditions used is independent of pH (range 5-10) and of temp. (18-27 c.). The rate of reaction with II depends on both, but decreases rapidly between pH 8-9. At pH < 8 at 27°c., the reaction with II is three times as fast as that with I, but, at about pH 9, the latter becomes the faster, which explains the similarity in the effects produced using a one or two bath process. Shrinkresist effects can be produced by means of ozone, but depend critically on the moisture content of the wool: between 0 and 13%, the effect gradually disappears, but at 17% it suddenly increases again, 20% being optimal as regards effect and least damage. H. E. N. regards effect and least damage.

Shrink-resisting Wool Yarn with Sodium Hypo-chlorite. K. Wojatschek. Melliand Textilber., 35, 64-65 (Jan. 1954).

The following method is preferred—the yarn is treated at room temp. for 20 min. in (per litre) 3-6 c.c. of aq. NaOCI (0.5-1 g. active Cl) and 5-10 c.c. HCl, much water is then added slowly, and working is continued for a further 20 min. After washing off, the yarn is antichlored in 3-4 g. NaHSO<sub>8</sub> per litre and rinsed. Lists of dyes (FBy) which resist chlorination after dyeing are given.

Cross-linking Reagents for Wool and their Physiological Effects. P. Alexander. Melliand Textilber., 35, 3-9 (Jan. 1954).

Methods of testing the ability of a reagent to cross-link wool molecules are discussed and methods of cross-linking are reviewed, 34 references being listed. Supercontraction in phenol can be prevented by boiling wool for 1 hr. in dil. acid. Out of the cross-linking of the tyrosine and lysine residues by formaldehyde, a method for mothproofing wool permanently has been developed. In this, phenols are linked to keratin by means of formaldehyde. For crosslinking, epoxides, ethyleneimines, mustard gas, nitrogen mustards, and polyfunctional mesyloxy cpd. (e.g. glycol ester of methanesulphonic acid) are used as follows: 1 g. wool in 25 c.e. water or bicarbonate, borax, or acetate buffer is shaken with 0-003 mole of reagent (in soln. or suspension) for 1 day and allowed to stand for 2 days at  $40^{\circ}\mathrm{C}$ . Out of 16 polyfunctional alkylating reagents tested, the following are shown to be especially successful cross-linking reagents.

Some of the reagents cross-link the wool through its amino groups, which suggests that amino groups are not present as ammonium groups in wool. Aq. sodium diethyldithio-carbamate is shown to be a very effective cross-linking reagent, also 1:5-diffuoro-, dichloro-, and dibromo-2:4-dinitrobenzene from bicarbonate buffer. Some of the reagents used here originated in research on growth-inhibition and carcinogenic activity, which appears to correlate with ability to cross-link protein molecules. Not all cross-linking reagents are potentially carcinogenic and a list of inactive cpd. is given, which includes dieceyanates, isocyanate deriv., 1:5-dihalogeno-2:4-dinitrobenzenes, I and III. H. E. N.

Microbe-resistant Wool by Chemical Modification.
II — Paper-chromatographic Examination. H.
Zahn. Melliand Textilber., 35, 162-167 (Feb. 1954).
Samples of wool, chemically modified and tested for

Zahn. Melliand Textilier., 35, 162–167 (Feb. 1954). Samples of wool, chemically modified and tested for microbe resistance previously (J.S.D.C., 70, 99 (Feb. 1954)), are hydrolysed by heating 0·1 g. with 1 c.o. of 68·HCl for 24 hr. at 105°c. The HCl is removed and the hydrolysate is analysed paper-chromatographically, full details of the technique being given. From wool treated with epichlorohydrin, no lysine (I) could be obtained, probably because of alkylation. Very little histidine (II) and some cysteic acid (III) were identified. From wool acetylated with acetic anhydride, some III and an aminoacid, probably ornithine from arginine, were obtained. Dinitrophenylated wool contains only little tyrosine (IV) or I, but their dinitrophenyl deriv., and very little II. Nitrated wool yields no IV and, surprisingly, only traces of III. Wool treated with formaldehyde gives the Millon reaction, but the hydrolysate contains no IV, supposedly because of secondary reactions. Wool treated with Kaurit 140 (Basf) or butanediol bischloromethyl ether yields greatly reduced amounts of IV. Wool treated with 4:6-bischloromethyl-m-xylene in acetone—aq. NaHCO<sub>3</sub> soln. contains lanthionine (V), but no I. V can be identified and even isolated in good yield from wool warmed with acetone and aq. NaHCO<sub>3</sub> only at 65°c. Reduced wool treated with methyl iodide gives S-methylcysteine, with 1:2-dibromoethane SS'-ethylene-bis-cysteine. The hydrolysate of KCN-treated wool contains much V, that of NaOH-treated wool V and III.

Modern One-bath Waterproofing. W. Otto. Textil-Praxis, 9, 367-369 (April 1954).

Current German proprietary wax-aluminium and wax-zirconium dispersions are treated in a general article, illustrated with photomicrographs. Wetting agents and alkalis must be removed from the goods before proofing. If required, the proofs may be stripped with boiling mineral acid plus a non-ionic dispersing agent. Reference is made to the hydrostatic head and the modified Bundesmann tests.

S. R. C.

Deterioration of Cuprammonium-proofed Jute Fabric, W. G. Memillan, S. N. Basu, and P. N. Pal. J. Sci. Ind. Research (India), 12B, 558-562 (1953): Chem. Abs., 48, 5419 (10 May 1954).

Cuprammonium-proofed jute on prolonged storage loses 60% of its tensile and bursting strengths without any loss in Cu content or evidence of growth of micro-organisms. This deterioration is due to liberation of acid from (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> deposited in the jute as a by product. Addition of Na<sub>2</sub>CO<sub>3</sub> to the impregnating liquor obviates this defect.

Fire-retardant Finishes. R. Freitag. Melliand Textilber., 35, 283–284 (March 1954).

Various processes are outlined and two recipes based on chloroparaffin-antimony oxide-zinc borate are given.

Coating Synthetic Fibres. K. Kubitzky. Melliand Textilber., 35, 66-68 (Jan. 1954). Both Perlon and Orlon fabrics can be coated using poly-

Both Perlon and Orlon fabrics can be coated using polyesters together with isocyanates (e.g. toluylene diisocyanate), which react during drying at 80°c. to form a firmly anchored film. The great resistance of such films to a variety of agencies is demonstrated.

H. E. N.

#### PATENTS

Crease-resisting of Regenerated Cellulose. Phrix-Werke. BP 710,441

The material is impregnated with an aqueous solution of an aliphatic dialdehyde or aliphatic a-ketoaldehyde having a straight or branched chain of < 11C and, as an acid catalyst, an aliphatic aldehydic carboxylic acid or an aliphatic a-ketocarboxylic acid, thus enabling the impregnating liquor to be kept at pH 3·5, after which the material is dried and baked. C. O. C.

Permanent Patterned Mechanical Effects on Fabrics. Heberlein & Co. BP 710,167

A local goffered effect is applied and then the fabric completely impregnated with a resin precondensate, dried, friction calendered, baked, washed and dried. The goffered effect is unaffected by the subsequent calendering which glazes the ungoffered portions of the fabric. C. O. C.

Wet Chlorination of Wool. Ciba. BP 709,377 Modification of BP 675,137. Improved chlorination results if the temperature of the liquor is raised at least towards the end of the treatment to  $60^{\circ}\mathrm{C}$ . or so. C. O. C.

Control of Wool Shrinkage by Polyamides. Montelair Research Corpn. and Ellis-Foster Co. USP 2,644,773 The wool is treated with an organic solvent solution of a water-insoluble polyamide and then dried. C. O. C.

Water-repellent Finish Resistant to Washing. Ciba. BP 709,333

Fibres containing no organically bound sulphur are treated in an aqueous dispersion containing (a) a water-insoluble ester of a methylol melamine in which at least one but not all the methylol groups are esterified with an aliphatic or cycloaliphatic earboxylic acid of > 8C, the unesterified methylol groups being at least partly etherified with a lower aliphatic alcohol, (b) an emulsifying agent, and (c) an acid or potentially acid catalyst. Preferably the dispersion also contains paraffin wax or other water-repellent wax, especially one which is soluble in the ester at raised temperature. Finally the materials are dried and treated for 5–15 min. at 125–140°c. It is unnecessary to wash the materials after baking as there is no unpleasant smell and the treated materials' resistance to penetration and surface wetting by water is very high.

The water repellent (a) above is replaced by a water-insoluble ether of methylol melamine with an aliphatic alcohol of > 3C mixed with a water repellent wax.

C. O. C. Patterned Pile Fabric. T. Brandon and H. Robertshaw.

Yarn impregnated with an adhesive is wound upon mandrels and then brought into contact with a backing material coated with an adhesive so that the yarn adheres to the backing to form a pile, the mandrels being finally removed and the yarn, if desired, cropped or cut.

C. O. C.

Bubble Pressure Test. II—Application to a Study of the Scaling of Close Cotton Cloths when Wetted (VI p. 371).

Mercerisation of Acid-treated Jute Fibre (VI p. 371).

Relaxation Shrinkage of Woven Wool Fabrics during Soaking in Water and Hoffman Pressing (VI p. 371).

Dyeing and Finishing Polyvinyl Chloride Fibres—New Developments (VIII p. 373).

### XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Some Aspects of the Application of Dyes to Paper. F. Parrott and K. W. Grundy. Proc. Tech. Sectn. Brit. Paper Board Makers' Assocn., 35, 15-30 (Feb. 1954). The selection of dyes, their evaluation in the laboratory, and the preparation of dye recipes for the mill are discussed, together with the use of colour-measuring instruments in the paper industry, and dyeing technique including mixing different classes of dyes, dye solubilities, matching to shade, and the causes and control of shade variation in the paper.

S. V. S.

Existence of a >C(-O-)<sub>2</sub> Chromophore—Ultraviolet Absorption of Cellulose. W. Berndt. Monatsh. Chem., 85, 387-392 (April 1954).

The possibility that the group COois a chromophore, which had previously been discussed by Mohler (Helv. Chim. Acta., 23, 1200 (1940)), is now investigated in the light of recent observations on the ultraviolet absorption behaviour of cellulose. No decisive proof, however, may yet be given from the data available.

H. H. H.

Detection of Free Hydroxyl Radicals during the Oxidation of Cellulose in Air. R. Holden. Nature, 173, 539 (20 Mar. 1954).

Cellulose fabric was impregnated with a solution of neutral sodium formaldehyde-sulphoxylate containing sodium naphthalene-1-sulphonate. After storage in a moist atmosphere, the fabric was made alkaline and treated with diazotised sulphanilic acid. A pink colour developed which is ascribed to coupling of the diazonium compound with a naphthol derived from the naphthalene sulphonate, hydroxyl groups being introduced into the naphthalenesulphonate as free hydroxyl radicals. No evidence of oxidation of cellulose, or of the presence of free radicals, has been obtained when sodium bisulphite, sodium formaldehyde bisulphite, or alkaline sodium formaldehyde sulphoxylate are oxidised on cellulose. Neutral sodium formaldehyde sulphoxylate is oxidised on glass fibre without formation of hydroxyl radicals.

W. R. M.

PATENTS

Sized Paper and new Sizing Agents. American Cyanamid Co. BP 709,979

Sized paper is prepared by adding to a dilute aqueous suspension of cellulosic papermaking fibres a small quantity (0·1-5%, preferably 0·1 to 3%, of the dry weight of the fibres) of the condensate of 1-2 mol. of epichlorhydrin with a total of 1 mol. of one or more unsat. or sat. aliphatic primary amines containing > 15C, forming paper from the treated suspension, and heating the paper at pH > 7 to dry it and develop the sizing properties of the condensate. 2-30% by weight of an alkaline filler, e.g. CaCO<sub>3</sub>, and an aqueous dispersion of a starch, gum or hydrocarbon wax, sufficient to deposit on the fibres 0·1-3% by weight of starch, etc., may be added in finely divided form after the condensate.

S. V. S.

Anti-sticking Cellulosic Sheets or Films. American Viscose Corpn. BP 710,331

A transparent, non-fibrous, stick-resistant film cast from an aqueous, alkaliné, cellulosic solution has a size on it comprising a colloidal inorganic water-insoluble solid material, e.g. colloidal silica, and a non-ionogenic, selfemulsifying, lubricious substance, e.g. a partial ester of a polyhydric alcohol and a higher fatty acid having at least 8 carbon atoms. The self-emulsifying substance may be a condensate of glycerol monostearate with ethylene oxide, with 1 to 100, e.g., 50 ethylene oxide units per molecule.

8. V. S.

Paper Coating Composition. National Cash Register Co. BP 710,597

A paper coating composition comprises a highly adsorbent substance, e.g. attapulgite (wholly or partially), an alkali metal silicate, e.g. sodium or potassium silicate, and another adhesive, e.g. a latex such as a butadiene-styrene copolymer, a styrene-isoprene copolymer, polychloroprene, or a methyl acrylate-acrylonitrile copolymer. The dry weight of the silicate used is from 1% to 20% of the dry weight of the adsorbent substance.

Water-resistant Coatings for Paper. Albright & Wilson. BP 710,792

A water-resistant coating colour comprises a slurry containing clay, natural or modified starch, a urea-formaldehyde resin and ammonium metaphosphate. The ammonium metaphosphate weighs 0.05% to 0.50% of the slurry weight; the starch, from 20% to 25% of the weight of the clay; and the resin, from 15% to 25% of the weight of the starch. The slurry contains 40% by weight of solids, and a viscosity reducer e.g. sodium silicate or sodium or ammonium phosphate glass. The colour is applied to paper or board and dried by applying heat.

Ultraviolet Absorption Spectra of Silk Fibroin and Cellulose in Lithium Bromide Solution (VI p. 372).

### XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Rôle of Lanthionine in the Unhairing Immunisation Phenomena. W. G. Shaw and R. M. Lollar. J. Amer. Leather Chem. Assocn., 49, 291–299 (April 1954).

Lanthionine is formed in steer hides under unhairing conditions and is found in equal amounts both when lime alone acts on the hair or epidermal area and when lime-sulphide solutions are used. This tends to disprove the contention that lanthionine formation is the cause of immunisation. A mechanism is suggested wherein lime alone forms calcium proteinate which has a "case-hardening" effect and resists the penetration of reagents such as sharpeners. Addition of Na<sub>2</sub>S at the start increases the alkalinity and reduces lime solubility, leading to less calcium proteinate and thus to better penetration.

J. W. B.

Degradation of Collagen. A Method for the Characterisation of Native Collagen. A. Veis and J. Cohen. J. Amer. Chem. Soc., 76, 2476-2478 (5 May 1954).

Estimation by the dye-protein precipitation method of the polar functional groups on highly purified hide collagen and several mildly degraded collagens showed no difference in the maximum number of basic dye binding sites on each sample per unit weight, but the concentration dependence of the binding is clearly related to the extent of degradation. Less than half of the theoretically available anionic groups are available as dye binding sites in the undegraded material. These become available in stages as the extent of degradation is increased. The thermodynamic functions  $\Delta F^\circ$  and  $\Delta S^\circ$  have been evaluated for the combination of the undegraded protein with Orange-G and Safranin-O. C. O. C.

Effect of Alkali Treatment of Collagen on the pH-Swelling Curve of Collagen and of Gelatin Products prepared from it. W. A. Loeven. J. Soc. Leather Trades Chem., 38, 117-126 (April 1954).

Leather Trades Chem., 38, 117-126 (April 1904).

The cause of the lowering of the isoelectric point of collagen, and of gelatin prepared under the action of alkali is investigated, and the prevailing view that the cause is a hydrolysis of amide groups of the partially amidated glutamic and aspartic acids is confirmed. A titration curve for a commercial sample of pigskin gelatin indicates that it can be regarded as solubilised collagen.

J. W. B.

### Solubility of Feather Keratin. A. M. Woodin. Nature, 173, 823-824 (1 May 1954).

Results of X-ray analysis, dialysis, and chemical reactions of solutions and products obtained from the dissolution of feather keratin in urea-bisulphite solutions are presented, and indicate that the keratin monomer has a mol. wt. of 10,000.

J. W. B.

### XIII- RUBBER; RESINS; PLASTICS

### Swelling Equilibria of Cross-linked Polyvinylpyrrolidones, J. W. Breitenbach and A. Schmidt. Monatsh. Chem., 85, 52-68 (Feb. 1954).

The preparations of cross-linked polyvinylpyrrolidones of limited swelling capacity are described from the mixed polymerisation of N-vinylpyrrolidones with diallyl and diethyleneglycol diallyl carbonate. The swelling equilibria of the cross-linked products are both theoretically and experimentally investigated as follows—(1) influence on swelling in water by carboxyl groups present. (2) swelling equilibria in 20 different liquids. (3) temperature dependence of swelling in 5 liquids. (4) swelling equilibria in water-dioxan mixtures. (5) swelling equilibria in aq. soln. of urea, cane sugar, methylene blue, cosin, inorganic salts (K<sub>2</sub>SO<sub>1</sub>, CuSO<sub>1</sub>, Na<sub>2</sub>PO<sub>2</sub>), and iodine. The enthalpies of mixtures of monomeric N-vinylpyrrolidones with H<sub>2</sub>O, CHCl<sub>2</sub>, and dioxan are measured. The swelling equilibria and their temp, dependence are in satisfactory agreement with the enthalpy data. Polyvinylpyrrolidone gels containing dissolved iodine afford additional cross-linking via formation of subsidiary valencies.

#### PATENTS

### Pile-like Rubber Surfaces. United States Rubber Co. RP 710.182

Material with a pile-like rubber surface is made by spraying on to a supported surface, from one or more spray guns, a mixture of coagulated and uncoagulated rubber particles, coarser particles being sprayed first if more than one spray is used. By adjusting the proportions of coarse and fine, and of coagulated and uncoagulated, rubber particles, and by having an electrode system above and below the spraying zone which permits charges of opposite polarity to be applied, the rubber layer is built up in discrete zones, thus giving a pile-like appearance.

J. W. B.

### Polyvinyl Chloride Stabiliser. Standard Oil Development Co. BP 702,848

Polyvinyl chloride and other chlorine-containing resins are stabilised against degradation by heat by adding 0·1-5·0% of a bis-hydroxyphenyl- or bis-hydroxyalkylphenyl-sulphide, sulphoxide or alkane of 3-7 C atoms, e.g. diphenylol propane. E. C.

### Metallised Plastic Foil. J. P. McGirr and T. G. McGirr. USP 2,644,761

Material looking like metal foil, of high tensile strength and moisture-proof, is obtained by forming a doughy mass of plastic material containing a large amount of metallic flakes into a film at controlled temperature. C. O. C.

### Reducing Weak Spots in Resinous Film or Sheeting. Union Carbide & Carbon Coron. BP 710.440

Union Carbide & Carbon Corpn. BP 710,440
Weak spots in resinous (e.g. vinyl resin or polyethylene)
film or sheet caused by the presence of electrically conductive particles are reduced or eliminated by subjecting
the film to a high voltage electrical discharge to burn or
otherwise disrupt the particles, leaving holes that are
eliminated by calendering. The electrical discharge may be
applied at one or more points close to the film before it
passes through the last nip of the calender (the bottom roll
being earthed) and varies from 4,000 volts for a four mil.
film of vinyl resin to 20,000 volts for a 50 mil. film. A threeroll calender may be employed, with an off-set roll adjacent
to the earthed top roll, plastic material being fed into the
nip, and a parallel electrode being in close proximity to the
top roll. Another electrode may be located close to the
film as it passes round the second roll, which is also earthed.
S. V. S.

### Embossing and Colouring Polyvinyl Materials. Dunlop Rubber Co. BP 710,085

A leaf of a vinyl polymer in which is dispersed a colouring matter and which softens at low temperature is applied to the vinyl material and a hot die impressed therein so as to fuse the coloured leaf to the polyvinyl material. C. O. C.

### XIV- ANALYSIS; TESTING; APPARATUS

### Anomalies in the Colorimetric Determination of pH.

L. Marcou. Textil-Rund., 9, 128-141 (March 1954).
Salts, proteins, adsorbents, and, particularly, surfaceactive agents (I) can alter the pH at which the colour change of an indicator (II) occurs, often by more than one unit. The following cases were investigated—(I) Anionic I (Na lauryl sulphate) + anionic II (Methyl Red) - error in pH determination increases with concn.; the colour change moves to the alkaline side. (2) Cationic I (lauryltrimethylammonium bromide) + cationic II (Neutral Red)— error in pH determination increases with concn.; the colour change moves to the acid side. (3) Nonionic I (polyethoxylated octylphenol and lauryl alcohol) + anionic II (Methyl Red and Thymol Blue) - error in pH determination increases with concn.; the colour change moves to the alkaline side. (4) Nonionic I (polyethoxylated octylphenol) + cationic II (Neutral Red) - error in pH determination increases with conen.; the colour change moves to the acid side. (5) Cationic I (lauryltrimethylammonium bromide) + anionic II (Methyl Red)—error in pH determination increases until the critical concn. (begin of micelle formation) is reached and then decreases; the colour change moves to the acid side. (6) Anionic I (Na lauryl sulphate) + cationic II (Neutral Red)— error in pH determination increases until the critical concn. is reached and then decreases; the colour change moves to the alkaline side. Indicators should therefore not be used for the determination of the pH of soln. containing surface active agents. It is shown that Pinacyanol chloride can be used to find the critical concn. of I as, in water, it exists in a dimeric form different in colour from the monomeric, present in organic solvents or micelles of I. 27 references. H. E. N.

### Use of Ethylenediaminetetra-acetic Acid in Analysis. H. Flaschka. *Textil-Rund.*, 9, 77–82 (Feb. 1954). The principles underlying the use of ethylenediaminetetra-

The principles underlying the use of ethylenediaminetetraacetic acid are reviewed and specific examples are given. 28 references. H. E. N.

### Spectrophotometric Method for Determination of Urea. G. W. Watt and J. D. Chrisp. Anal. Chem., 26, 452-453 (March 1954).

The yellow colour produced when p-dimethylaminobenzaldehyde is added to urea in dil. HCl soln., shows an absorbance max. at  $420~\mathrm{m}\mu$ ., with validity of Beer's law up to conon. of urea of  $320~\mathrm{p.p.m.}$  The described procedure shows a relative error of 1% over the optimum range of  $50-240~\mathrm{p.p.m.}$  urea. Hydrazine and semicarbazide interfere, and a method is given for their removal;  $\mathrm{NH_4^+}$  does not interfere aignificantly.

### New Test for Scourability of Spinning Oil. A.A.T.C.C. Committee on Processes of Continuous Wool Scouring. Amer. Dyestuff Rep., 43, P263-P265, P269 (26 April 1954).

Two methods are described for evenly applying oil to small amounts of test yarn. Firstly, dry-cleaned wool yarn is dipped in a 10% solution in toluene of oil to be tested, centrifuged, and dried, and secondly, single end padding with a laboratory padder containing the oil solution in toluene, followed by drying. A continuous scour procedure is then adopted.

J. W. B.

### Chromatographic Method for Identifying Synthetic Detergents. J. Blandin and R. Sesalme. Bull. mens. ITERG, 8, 69-73 (1954): Chem. Abs., 48, 6146 (25 May 1954).

Chromatograms produced on filter paper strips by dividing them in an aqueous solution containing Fluorescein, Fuchsin, Methylene Blue and Blue for Silk 6B, show characteristic changes of colour according to the detergent added to the solution. The range of colours obtained with 20 different detergents is given. C. O. C.

### Colorimetric Determination of Anionic Detergents. F. J. Loomeijer. Anal. Chim. Ana, 10, 147-150

(1954): Chem. Abs., 48, 6146 (25 May 1954).

Many types of anionic detergents can be determined colorimetrically by means of their action on a protein—Bromocresol Purple complex; within limits, the amount of free dye liberated from the complex is proportional to the amount of detergent added. The accuracy, ca. 4%, is not

as good as that of methods based on rosaniline, but the protein-complex method is not restricted to long-chain alkyl sulphates, and so can be applied to many more anionic detergents than can the rosaniline method.

Antistatic Agent Evaluation. W. Sprenkmann. Melliand Textilber., 35, 93-95 (Jan.), 307-308 (March 1954).

II - Modifications to the apparatus of Hayek and Chromey (J.S.D.C., 67, 295 (1951)) are described and these lead to more reproducible results. Measurements by the much simpler procedure previously described (J.S.D.C., 70, 330, (July 1954)) correlate surprisingly well.

III-Formulæ are derived for Schwenkhagen's rotating static voltmeter, adapted for the measurement of static electricity on fibres and surfaces. The need for thorough humidity control in making measurements of static electricity is emphasised. H. E. N.

Determination of Fluoride Ion using a Monohydroxy-azo Dye-Thorium Lake. J. L. Lambert. Anal Chem., 26, 558-560 (March 1954).

Fluoride ions, which form very stable complex ions with Zr and Th, react to displace the dye mol. from chelation lakes of Zr and Th with 1:2-dihydroxyanthraquinone dyes the conon. of dye released being measured spectrophoto-metrically. Amaranth is found to be the most satisfactory dye for this purpose.

er Chromatography of some Substituted Naphthaquinones. T. Sproston and E. G. Bassett. Anal. Chem., 26, 552–553 (March 1954). Paper

Mixtures of substituted naphthaquinones are resolved by running two-dimensional chromatograms for 15-18 hr. with amyl alcohol-pyridine-water (3:2:1.5); the dried chromatogram is developed by spraying with 5% aq. NaOH. The max. variation for any one compound is  $\pm\,0.04$   $R_f$  value. A table is given which shows the  $R_f$  values and colours of 37 substituted and unsubstituted quinones. The experimental procedure is detailed.

Analysis of the Copper Compound, Harmful to Rubber, present in Phthalocyanine Blue. 8. Minatoya, I. Aoe, and H. Ishiyama. J. Soc. Rubber Ind. Japan, 26, 76-82 (1953): Chem. Abs., 48, 6155 (25 May

In establishing the Japanese industrial standard for Phthalocyanine Blue, the maximum of free Cu was specified as <0.1%. This amount was confirmed to be valid by carrying out ageing tests of a rubber mix containing Phthalocyanine Blue.

Simplified Procedure for Separating Porphyrins from Urine by Paper Chromatography. L. M. Corwin and J. M. Orten. Anal. Chem., 26, 608-609 (March 1954).

Porphyrins are adsorbed quant. on lead salts pptd. by the addition of lead acetate soln. to urine, the ppt. separated centrifugally, and the porphyrins are eluted with HCl (12-15%) which is subsequently evaporated under reduced pressure over NaOH pellets. The residue is taken up in NH4OH (10N.) the resulting soln. being subjected to paper chromatography (by a test-tube method which is described in full). The paper is developed with a 1:1 described in full). The paper is developed with a 1:1 mixture of 2:4- or 2:6-lutidine and water, and the air-dried chromatogram is examined and marked under ultraviolet radiation.

Identification of Dyes in Lipsticks by Paper Chromatography. J. Deshusses and P. Desbaumes. Mitt. Gebiete Lebenom. Hyg., 44, 500-507 (1953): Chem. Abs., 48, 6079 (25 May 1954).

Dissolve the lipstick in warm 50% acetic acid, purify the filtrate with petroleum ether, evaporate, dissolve in 50% alcohol and subject the centrifuged solution to paper chromatography. C. O. C.

Comparison of Some Methods for Evaluation of Chalking Resistance of Titanium Dioxide Pig-ments. E. Lund and C. F. Weider. F.A.T.I.P.E.C. (Féd. assoc. tech. inde. peintures, vernis, émaux et encres imprimerie Europe Continentale), 2° Congr., 286-291 (1953); Chem. Abs., 48, 5518 10( May 1954).

Some agreement occurs between results obtained with laboratory methods (e.g. Weather-o-meter, mandelic acid and Lithol Red fading tests) and results from natural exposure. The latter is dependent on weather and limited to the summer months. To speed up chalking the pigment volume concentration was increased to ca. 33% and an alkyd paint was made grey by adding carbon black. Chalking is attributed to photoreduction and the anatase crystal unit is more labile than the rutile crystal. With finger rubbing it is difficult to distinguish between heavy chalking paints. 500 hr. in the Marr Weath-o-meter correspond to ca. 2200 hr. (3 months) outdoor exposure, but only for break down of the film by chalking, not for cracking and scaling. Reproductibility of the mandelic acid test is not too good and the average has to be taken of several tests. With the Lithol Red fading test better results are obtained with "free chalking grades" than with the more chalking-resistant qualities. Chalking is heavier and starts earlier with linseed oil paints as the oil has less resistance to photochemical breakdown. Alkyd paints are resistance to photochemical breakdown. Alkyd paints are more suitable for testing purposes. The grey alkyd paint is ground on a 3-roller mill to ca.  $25\mu$ , then 2 coats are brushed on an iron panel  $280 \times 190 \times 2$  mm. and exposed on a roof in a  $45^\circ$  angle facing south from May-October. Photochemical reaction caused by sunlight leads to formation of a CO<sub>2</sub> gas-film between the pigment grains and binder, and after having lost its adhesion the TiO<sub>2</sub> particles come off the paint like chalk, masking the surface and giving increased light reflectance. The chalking rate can thus be followed colorimetrically.

Stereomicroscopes. E. von Gizycki. Melliand Textilber.,

35, 263-264 (March 1954).

A microscope with one objective and two eyepieces together with a suitable camera is described. H. E. N.

Standard Method of Test for Fluidity of Cotton, Rayons, and Cellulose Acetate in Cupram-monium Hydroxide Solution. Tentative Textile Standard No. 25. J. Textile Inst., 45, 818-826 (April 1954).

Bubble Pressure Test for Measurement of Pore Size in Fabrics. I—Development of Test. J. Lord and H. M. Taylor. J. Textile Inst., 45, T371-T379 (May 1954).

An instrument is designed and a technique devised for measuring the equivalent pore size of textile fabrics. It is based on the determination of the pressure to force air through the pores of a specimen of fabric which is com-pletely wetted-out by a supernatant liquid of known surface A 3 in, square of fabric, conditioned for 24 hr. at 65% R.H. and 68°r., is totally immersed and scaked in test liquid for 3 min. It is clamped over a testing head and sufficient liquid poured on the surface to cover the specimen after it has bulged slightly from pressure to be exerted from underneath. Air pressure is applied to the underside, raised at 20 cm. head of water per minute, and noted when the third stream of bubbles emerges. White spirit is most suitable as the liquid.

The equivalent radius of the third largest pore in the specimen, rbp, is calculated from the formula-

$$r_{
m bp} = \frac{2S}{P_{
m b} I\!\!I} \times 10^4 \mu$$

(S= surface tension of white spirit, in dynes/em.;  $P_{\rm b}=$  pressure; and  ${\it g}=$  the gravitational constant). The effects of numerous variables are studied.

Fault Identification in Fabrics—II. A. Wylezieh. Textil-Praxis, 9, 358–363 (April 1954).

A number of unrelated faults, together with their histories and modes of investigation, are discussed. The Krais-Viertel test was used in certain instances for the characterisation of sulphuric acid damage. 8. R. C.

### Quantitative Determination of Amino Acids using Monodimensional Paper Chromatography. J. F. Roland and A. M. Gross. Anal. Chem., 26, 502-505 (March 1954).

A simple method suitable for routine use has been developed in which 2-butanol-3% ammonia (3:1) and 72% phenol are used monodimensionally, 16 amino acids being sufficiently well resolved for their quant. determination by the direct photometry of their ninhydrin spots. Resolution requires ca. 48 hr. Tryptophan is not satisfactorily resolved but may be so by use of 2-butanol-95% ethanol-6% NH<sub>4</sub>OH (100:25:35) in 24-40 hr., and histidine requires likewise either development with Pauly's reagent after resolution by 2-butanol-3% ammonia on a separate sheet, or resolution with an alternative solvent system such as 2-butsnol-acetic acid-water (120:15:25) for 64 hr. Results obtained by applying the method to a mixture of 15 amino acids designed to simulate bovine serum albumin hydrolysate agree well with previously reported values. The methods are fully described, a typical chromatogram is shown, and typical standard curves for the determination of 10 amino acids are reproduced.

J. W. D.

### Differentiation of Polyamide Fibres. H. Ludewig. Textil-Praxis, 5, 463-464 (May 1954).

The melting points of nylon, Perlon, and Eftrelon, observed in liquid paraffin to exclude air, are  $255^{\circ}\mathrm{c}$ ,  $215^{\circ}\mathrm{c}$ , and  $238^{\circ}\mathrm{c}$ . respectively. Perlon and Eftrelon dissolve in cold  $4\cdot4\mathrm{N}\cdot\mathrm{HCl}$ , nylon requiring heat. On dilution with water, the solutions become turbid. In  $8\cdot3\mathrm{N}\cdot\mathrm{H_2SO_4}$ , Perlon and Eftrelon dissolve at room temperature, nylon again requiring heat. After prolonged boiling in  $8\cdot3\mathrm{N}\cdot\mathrm{H_2SO_4}$  (liquor ratio 20:1) Eftrelon shows turbidity, whilst Perlon solutions remain clear.

### Chromatographic Method for Examining the Dyeing Properties of Wool Dyes. H. Egli and M. Perrig. Textil-Rund., 9, 61-69 (Feb. 1954).

A column, 7 cm. long, is constructed of 42 discs of woo felt (= 3 g.) held inside the chromatographic tube. This is situated in a steam jacket, which maintains an internal temp. of 97-98°c. The wool is wetted by a blank soln., dye (10 mg., i.e. 0·33%) is then added to the soln. and allowed to be adsorbed. The chromatogram is developed until dye appears in the cluate. The column is dissected and the disc of deepest colour noted by its number (I) from the top of the column. A very pretty, easily understood picture of the equalising properties of the dye is obtained. Within the limits of speed of flow used, the speed of migration of the dye (II) remained proportional to it. II is independent of time. With increasing pH of the liquer, II increases more and more rapidly. When pH is plotted against I, each dye gives a characteristic line. From such graphs, it can be seen e.g. that Xylene Fast Blue P at pH 5-5-4 has equalising properties comparable to those of Azo Rhodine 6B at pH 2·4. This chromatographic method enables dyes to be distinguished which do not show different properties in the usual migration test. It is shown that 10% Na,804 greatly increases II of Azo Rhodine 2G at pH 2·8, increases that of Xylene Fast Blue PR at pH 4·7, and decreases that of Xylene Brilliant Cyanine G at pH 6·7, added salt slightly increases II. The helpful effect of equalising agents is demonstrated.

## Fastness Evaluation. G. Schwen, J. Eisele, and S. Hafenrichter. Melliand Textilber., 35, 281–283 (March 1954)

The E.C.E. prescribed certain depths for dyeings of various hues as standard depths for testing the fastness properties of dyes. Dyeings of depths of 2/1, 1/3, 1/6 and 1/12 of these standard depths are necessary also, but no specifications exist for their production. For ten hues, the amounts of dye required under standard conditions to produce such dyeings have now been determined. The dyes used show good exhaustion and the amounts of dye required are near those that would be expected if the degree of exhaustion were independent of conen.

H. E. N.

Spectrophotometric Modification of the Cuprous Oxide Method for Determining Sugars. M. E. Hobbs and F. L. Layton. Anal. Chem., 26, 585–586 (March 1954).

Cuprous oxide pptd. from Fehling's soln. is dissolved in hot HNO<sub>3</sub> (4m.) and the ethylenediamine complex is formed. The absorbance of the resulting soln. is measured at 550 mµ. The method is more rapid than the conventional gravimetric finish, and has been found to be of satisfactory precision if the absorbance is arranged to be > 0·1.

J. W. D.

### Hydrolysis of Starch as indicated by Congo Red. B. Carroll and J. W. Van Dyk. J. Amer. Chem. Soc.

75, 2506–2511 (5 May 1954). Adsorption of Congo Red by amylose and amylopectin is indicated by a spectral change. At pH 7 adsorption of dye is accompanied by 21% decrease in optical density at  $\lambda=400$  m $\mu$ s. Considerably larger spectral change is seen at a pH in the region of the pK of the dye. The specific adsorption depends upon the type of starch and its molecular weight. The enzymic activity of pancreatic amylase is determined by the spectral change in a starch–Congo Red solution. Compared with the starch–iodine method the colour change is much slower especially in the early stages of the hydrolysis. The point at which a hydrolysed starch just fails to yield a red with iodine still produces marked spectral change with Congo Red. C. O. C.

## New Quantitative Reaction for Lignin. S. N. Beketovskii. J. Anal. Chem. U.S.S.R., 9, 112 (March-April 1954).

Lignin-containing material gives a green coloration when spotted with cupric nitrate soln. (2n. or conc.). A. E. S.

## Methods of Analysis of Leather Finishing Materials. American Leather Chemists Association. J. Amer. Leather Chem. Assocn., 49, 258-262 (April 1954).

Standard methods of estimating total solids and ash in leather finish, cellulose nitrate in finish, and the flexibility, adhesion, and tackiness of finish on leather are given.

J. W. B.

# Measurement of Colour, Gloss, and Haze of Plastics. H. K. Hammond and G. W. Ingle. Symposium on Plastics Testing, Present and Future, ASTM Spec., Tech. Pub. No. 132, 25-31 (1953): Chem. Abs., 48,

5543 (10 May 1954).

The capabilities and limitations of tests for colour, gloss and haze are discussed. Adaptations of the test methods are described.

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### PATENT

# Identification or Analysis of Substances by Radio-frequency Resonance. Lantson. BP 710,373 An apparatus is described whereby liquid mixtures, and solutions of solids, may be analysed, e.g. for control purposes in petroleum fractionation, by generating electromagnetic waves within the radio frequency band, submitting the substance to their action, and measuring the frequency at which maximum resonance is produced or the maximum energy absorption by the substance at resonance. Reference to previously obtained calibrations, on liquids of known composition, enables rapid assessment of the proportions. J. W. B.

Relaxation Shrinkage of Woven Wool Fabrics during Soaking in Water and Hoffman Pressing (VI p. 371). Dynel "Inner Structure" (VI p. 372).

Wet Processing of Recovered Wool (VII p. 373).

Matching Colours (VIII p. 373).

Thickening Agents—Measurement of the Properties of Pastes (IX p. 374).

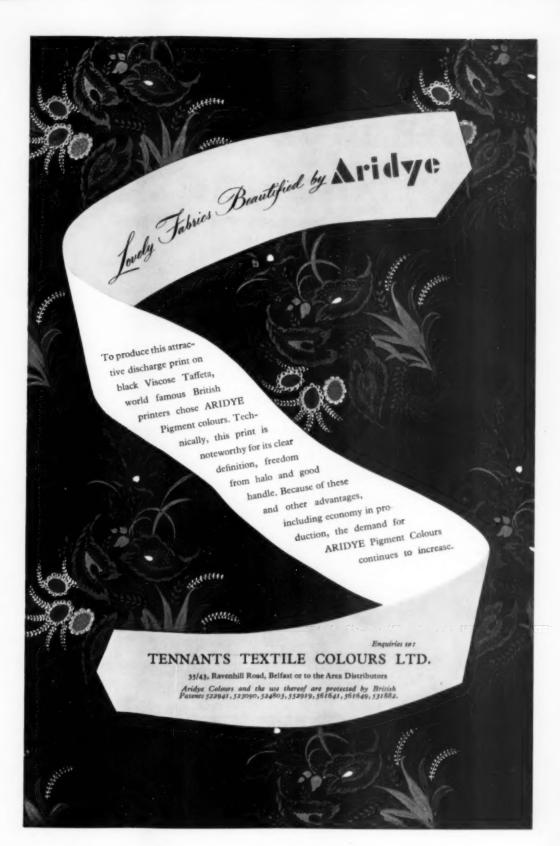
Better Mercerising for Improved Lustre (X p. 375).

Microbe-resistant Wool by Chemical Modification. II— Paper-chromatographic Examination (X p. 376).

Detection of Free Hydroxyl Radicals during the Oxidation of Cellulose in Air (XI p. 377).

### XV-MISCELLANEOUS

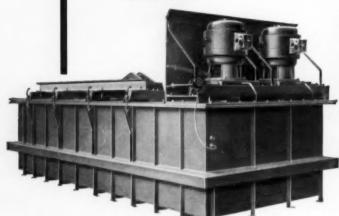
### Historical Notes on the Wet-processing Industry. IV— The Rôle of Robert Hooke. S. M. Edelstein. Amer. Dyestuff Rep., 43, 315-316 (10 May 1954).



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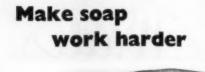
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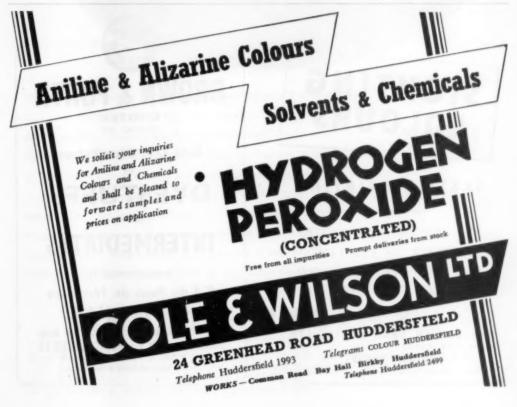


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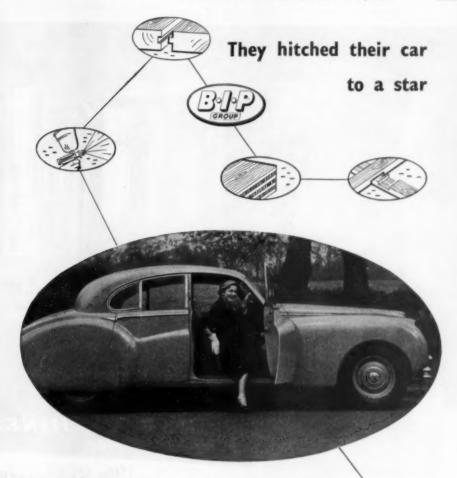
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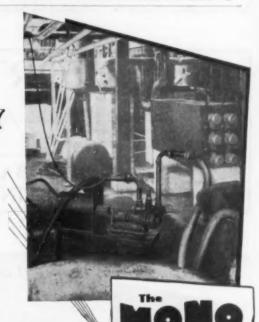
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